EDGEWOOD

CHEMICAL BIOLOGICAL CENTER

U.S. ARMY RESEARCH, DEVELOPMENT AND ENGINEERING COMMAND

ECBC-TR-690

EVALUATION OF GASMET™ DX-4015 SERIES FOURIER TRANSFORM INFRARED GAS ANALYZER

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DEPARTMENT OF PROGRAM INTEGRATION

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14. ABSTRACT
The Mobile Laboratories and Kits Team, U.S. Army Edgewood Chemical Biological Center, was funded by the Joint Product Manager for Consequence Management to perform an instrument validation and verification test of a Gasmet™ DX-4015 Series Fourier Transform Infrared (Gas Analyzer. This instrument was chosen for use in the National Guard Bureau's Analytical Laboratory System (NGB-ALS) Increment I Analytical Revision. The NGB-ALS wanted documentation on the instrument's performance and ability to identify gaseous chemical warfare agents, toxic industrial chemicals, and toxic industrial materials.

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PREFACE

The work described in this report was authorized under Project No. 07P-0009. The work was started in October 2007 and completed in March 2008.

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EVALUATION OF A GASMET™ DX-4015 SERIES FOURIER TRANSFORM INFRARED GAS ANALYZER

1. INTRODUCTION

1.1 Background.

The Joint Program Manager Guardian (JPMG) is an organization within the Joint Program Executive Office for Chemical Biological Defense. The JPMG is comprised of three Joint Product Management (JPM) offices which provide distinct, but not unrelated, services and products. One of those offices is the JPM for Consequence Management (CM), formerly known as PM Weapons of Mass Destruction—Civil Support Systems, and supports the National Guard Bureau, U.S. Army Reserve and other response units through development, procurement and fielding of critical CBR incident protection and response capabilities. One of these responsibilities is the Analytical Laboratory System (ALS) used by the National Guard Bureau (NGB).

In 2007, the JPM-CM funded the Mobile Laboratories and Kits Team, U.S. Army Edgewood Chemical Biological Center (ECBC), to perform an instrument validation and verification test of a Gasmet™ DX-4015 Series Fourier Transform Infrared(FTIR) Gas Analyzer. This instrument was being considered for use in the NGB-Analytical Laboratory System (ALS) Increment I Analytical Revision. The NGB-ALS wanted documentation on the instrument's performance and its ability to identify gaseous Chemical Warfare Agents (CWAs) and volatile Toxic Industrial Chemicals/Toxic Industrial Materials (TICs/TIMs).

1.2 Objective.

The objective of this study was to evaluate the performance of the Gasmet™ DX-4015 FTIR system as an analytical technique for the identification of select gas phase CWAs (Table 1) and volatile TICs/TIMs (Table 2) that are gases at ambient room temperature or at temperatures from 20 (68 °F), 22 (71.6 °F), to about 24 °C (75.2 °F).

Table 1. CWAs

Hydrogen Cyanide
Phosgene
Phosphine
Chlorine

Chlorine was initially added to Table 1 as a check component to verify the manufacturer's claim that the instrument cannot be used to analyze chlorine as a target compound or compound of interest. However, chlorine was not tested as the instrument had no spectrum of chlorine loaded into the target library list and due to the manufacturer's claim that low concentrations of chlorine would damage the instrument.

Table 2. TICs/TIMs

Boron Trifluoride
Arsine
Phosgene
Phosphine
Ammonia
Carbon Monoxide
Sulfur Dioxide
Ethylene Oxide

2. EVALUATION PROCESS

Two instruments were selected and purchased. One instrument was subjected to two tiers of testing. Tier 1 testing consisted of pre- and post-ruggedness testing limit of detection (LOD) estimations for common target analytes. If the instrument passed the ruggedness testing, it was then subjected to Tier 2 performance testing with select CWA and TIC/TIM analytes. If the instrument failed the ruggedness testing, a determination was made on the extent of repair needed and the time involved. See Figure 1 for the evaluation process.

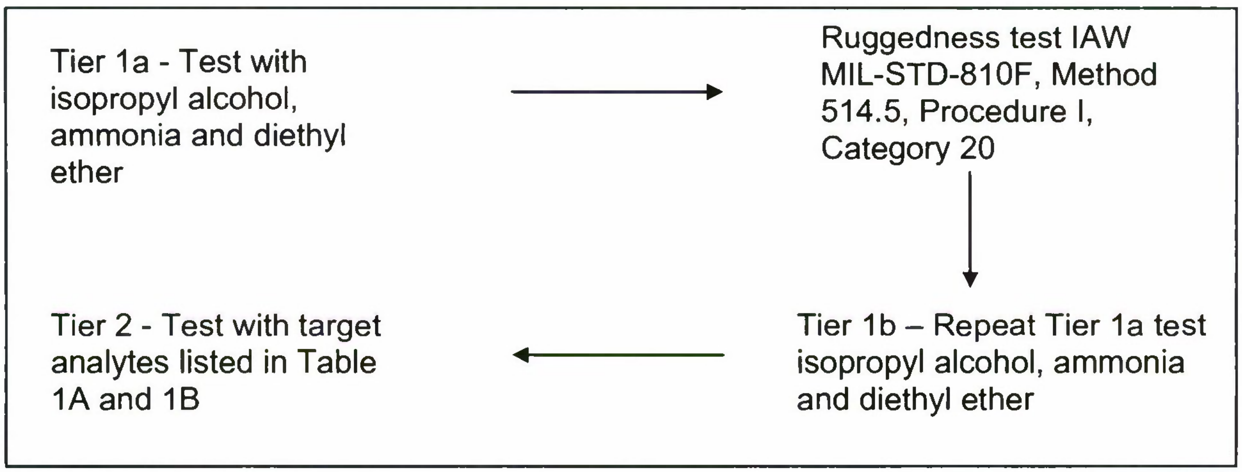

Figure 1. Evaluation Process of the Gasmet™ DX-4015 Series FTIR Gas Analyzer

Instrument test conditions simulated current ALS Increment 0 conditions. Briefly, unknown samples are collected in a "hot zone," packaged, decontaminated, and transported to the ALS for chemical analysis. Samples are initially placed in engineering controls where the sample container is opened and screened for CWAs and select agents followed by presumptive analyses by FTIR for CWAs and TICs/TIMs. Gas samples were taken and analyzed using

400 mL summa canisters. It was determined that the size of the summa canister had a greater affect on the method LOD and not the instrument LOD. The bigger the summa canister, the more completely the gas cell can be filled, the more accurate the analysis results are quantitatively.

During instrument verification testing, LODs, specificity, selectivity, and inter-run reproducibility and inter-operator reproducibility were evaluated for each gas analyte. The LOD was determined or approximated based on the lowest concentration of each gas analyte tested. Testing indicated that the LOD for each gas tested was below 1.0 ppm. The LOD was anticipated to be at or near 1.00 ppm. Testing indicated that the instrument was quite capable of detection limits <1.00 ppm but that would have required the purchasing of additional cylinders of diluted gas mixtures at lower concentrations. This would have required more time and more money and therefore was determined to not be feasible. The compounds were evaluated in only the gas phase at ambient or normal room temperatures and/or normal environmental conditions. The ability of the instrument to clearly identify each gas independent of the operator was also evaluated using a minimum of two operators.

2.1 Equipment and Devices.

The equipment and devices used for the instrument verification testing are listed below:

- Gasmet™ DX-4015 FTIR Gas Analyzer
- Chemical fume hood or glovebox
- Gas cylinders
- Flow meter
- Gas regulators
- Nitrogen generator
- Laptop Computer
- Summa Canisters

2.2 Materials.

The materials used for the instrument verification testing are listed below:

- 0.5 to 25 µg/mL gas mixtures and pure gas components of target analytes listed in Tables 1 and 2.
- Tygon and/or Teflon tubing
- Swagelok fittings
- Tedlar Bags
- Kimwipes
- Nitrile Gloves
- Miscellaneous gas sampling and tubing supplies
- 0.5% Sodium Hypochlorite Solution (decon)
- DI Water (decon)
- Acetone (decon)

2.3 Sample Receipt and Handling.

Samples for this validation study were prepared by Airgas Specialty Gases and Equipment. Sample gas preparation schemes were formulated in advance and reviewed by the Senior Chemist prior to preparation. Sample test gas cylinder preparation was performed by Airgas Specialty Gases and Equipment and documented by the operators in the sample preparation lab notebook. Testing consisted of 40 standard 33A size gas cylinders containing one target TIC/TIM gas analyte with nitrogen as the balance gas. The concentrations tested included the following:

- Ammonia at 0.7, 3.68, 8.31 and 20.9 ppm ± 10%;
- Arsine at 1.0, 5.02, 10 and 23.8 ppm ± 10%;
- Boron Trifluoride at 10.8, 24.9, 50.1 and 100.3 ppm ± 10%;
- Carbon Monoxide at 1.24, 5.296, 10.09 and 24.41 ppm ± 5%;
- Hydrogen Cyanide 1.02, 5.05, 10.3 and 24.9 ppm ± 10%;
- Ethylene Oxide at 4.0, 10.61 and 24.45 ppm ± 5%;
- Phosgene 1.0, 4.97, 9.95 and 24.9 ppm ± 10%;
- Phosphine at 1.0, 5.01, 9.96 and 24.9 ppm ± 10%; and
- Sulfur Dioxide at 0.986, 4.988, 10, 25.18, 49.98 and 99.83 ppm ± 5%.

The concentrations tested and verified were below the LOD levels defined and established in the Capabilities Production Document and are listed in Appendix A.

The cylinder was connected to the appropriate regulator with a 6 in. long section of ¼ in. silco-steel tubing, which had a quick-connect end. A similar section of tubing was connected to the sample inlet of the DX-4015. The quick connects were joined, and the valve on the cylinder was opened and adjusted to maintain approximately 30 psi on the regulator. The DX-4015 and computer were set to acquire data and the internal pump of the Gasmet DX-4015 was started. Approximately 50 to 60 analytical runs at 1 min a run were made from one cylinder. A 400 mL silco-steel summa canister was used by first pulling a vacuum on the cylinder via a quick connect and using the DX-4015's internal pump. At ambient temperature, a vacuum of about 15 mm Hg was pulled on the same summa canister for each analytical run. The 33A sample cylinder was opened and the summa canister was attached and opened, quickly drawing in the test gas sample. The summa canister was then transferred to the DX-4015 and attached to the quick connects. The instrument and computer were set to acquire data on the specific set of test gases and started. When using the 400 mL summa canisters, testing indicated that the size of the summa canister produced a concentration reading on the DX-4015 of approximately one third or, in some cases, 3 to 5 times less than the actual concentration of the gas in the cylinder (Table 2A). However, the DX-4015 accurately identified each gas in the 33A cylinder from the summa canister. This difference can be corrected by using larger summa canisters that would completely fill the gas cell of the DX-4015 long enough to get an accurate reading or employ a multiplication factor of 5 when collecting and analyzing samples using the smaller 400 mL summa canisters.

Table 3. Ambient Gas Analysis Using 400 mL Summa Canisters

	Phosgene Run #1	Phosgene Run #2	Phosgene Run #3	Hydrogen Cyanide Run #1
Initial Concentration	24.9 ppm	24.9 ppm	24.9 ppm	24.9 ppm
Measured Concentration #1	3.08	1.05	9.71	0.12
Measured Concentration #2	3.49	4.90	9.43	1.83
Measured Concentration #3	1.20	4.11	2.73	9.98
Measured Concentration #4	0.39	2.66	0.82	9.28
Measured Concentration #5	0.15	1.58	0.30	6.09
Measured Concentration #6	0.07	0.93	0.16	3.43
Measured Concentration #7	0.04	0.56	0.12	1.91
Measured Concentration #8	0.02	0.35	0.10	1.01
Measured Concentration #9	0.01	0.21	0.33	0.49
Measured Concentration #10	0.00 NP*	0.13	0.28	0.31

*NP - Analysis was not performed or taken because the canister had emptied before the next sample could be measured.

3. TECHNICAL APPROACH

The Gasmet™ DX-4015 FTIR (Figures 2 and 3) is a compact bench-top FTIR capable of analyzing gases directly.

Figure 2. Gasmet™ DX-4015

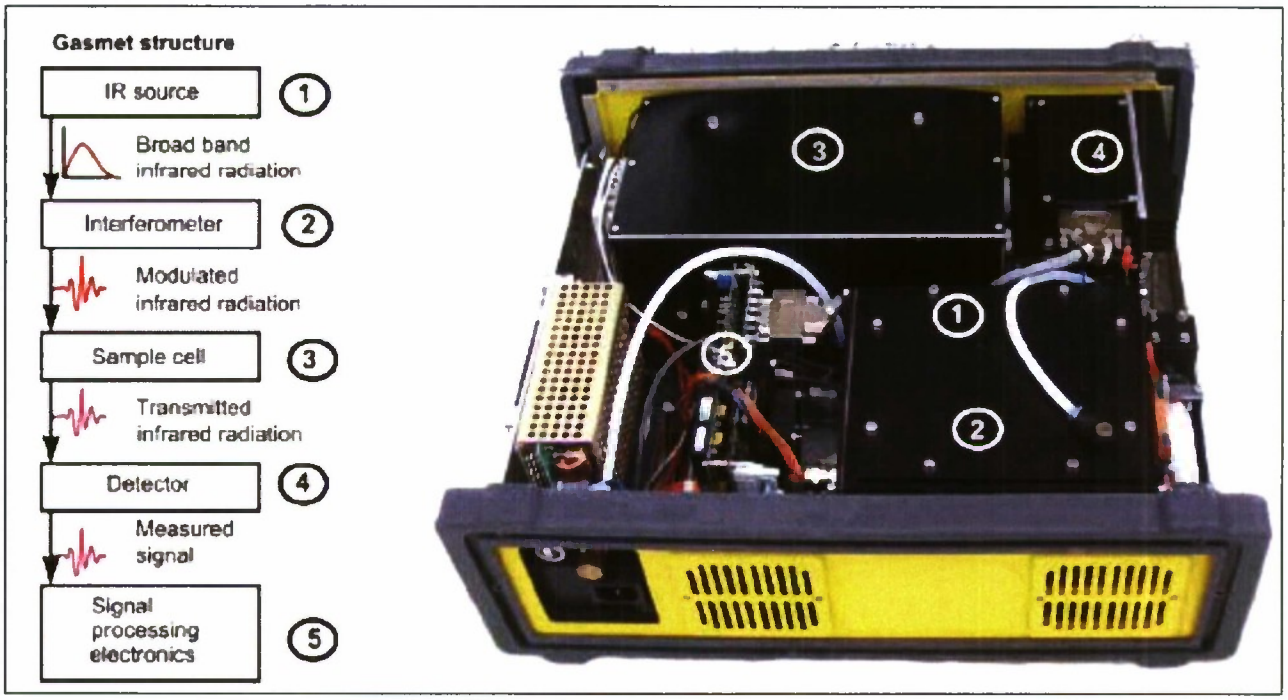

Figure 3. Basic Structure of the Gasmet™ DX-Series Analyzer

The detector measures the absorption spectra of gases that have been exposed to in-phase infrared radiation. An infrared laser provides the radiation that is focused on the sample gas introduced into a 1M cell. Emission radiation can be collected following a reflection or transmission process. Only one system is being considered and that system is a reflection system. Molecular bonds have multiple energetic states including a ground state and single or multiple excitation states. If light energy equivalent in frequency to the difference between the ground state and an excitation state strikes the molecule, it will be absorbed putting that bond into the excited state. Energy is subsequently emitted by the molecule taking it back to ground state. Measurement of the absorption frequencies of a sample produces a spectrum that can be used to identify functional groups and consequently structure. Fourier Transform Infrared can be used to analyze solid, liquid, and gas-phase samples. However, without a validated separation technique preceding FTIR analysis to provide selectivity, its utility for analyzing complex mixtures is limited. This instrument was tested against gas mixtures of Ammonia, Boron Trifluoride, Hydrogen Cyanide, Phosgene, Arsine, Phosphine, Carbon Monoxide, Sulfur Dioxide, and Ethylene Oxide in nitrogen and the instrument was able to differentiate each mixture.

FTIR spectroscopy was developed to eliminate the problems associated with dispersive instruments such as slow scanning and the ability to measure all the infrared frequencies simultaneously, rather than individually. FTIR spectrometry employs a very simple optical device called a Michelson interferometer (Figure 4).

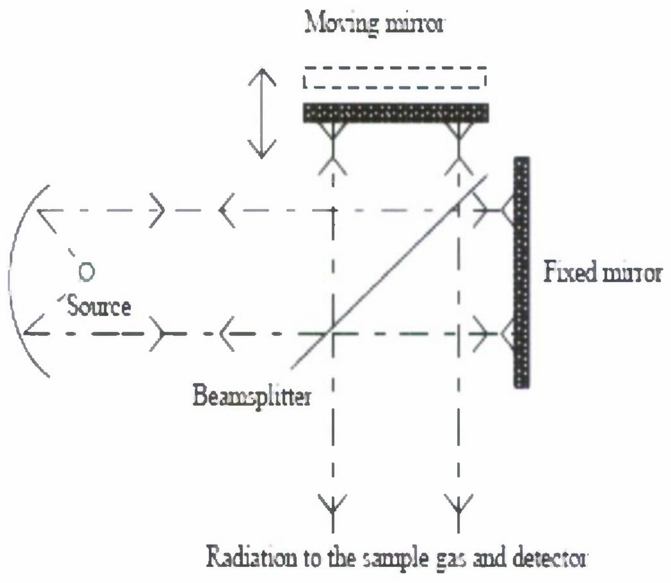

Figure 4. Michelson Interferometer

The interferometer consists of a source, beam splitter, two mirrors, a laser, and a detector. The beam splitter takes the incoming infrared beam and divides it into two parts or optical beams. One beam reflects off a flat mirror which is fixed in place. The other beam reflects off a flat mirror mounted on a mechanism that allows this mirror to move a very short distance, typically a few millimeters, back and forth away from the beamsplitter at a constant velocity. This velocity is timed according to the very precise laser wavelength in the system, which also acts as an internal wavelength calibration. The two beams are reflected from the mirrors and recombined at the beamsplitter. The beam from the moving mirror travels a different distance than the beam from the fixed mirror and because the path that one beam travels is a fixed length and the path of the other beam is constantly changing, an interference pattern is created when the beams are recombined. Some of the wavelengths recombine constructively and some recombine destructively. The resulting signal or interference pattern is called an interferogram, which has the unique property of every data point making up the signal having information about every infrared frequency coming from the source, or, in other words, the interferometer produces a unique type of signal which has all of the infrared frequencies "encoded" into it. This interferogram then goes from the beamsplitter to the sample where some energy is absorbed and some is transmitted.

The transmitted portion reaches the detector, which means that as the interferogram is measured, all frequencies are being measured simultaneously. This results in an IR spectrum and shows that the use of an interferometer results in extremely fast measurements.

3.1 Instrument Verification.

The DX-4015 FTIR system was verified (Figure 5) on a daily basis during the validation process. Prior to analyses, FTIR system performance was verified according to manufacturer's specifications (Shaker Test Report included in Appendix B).

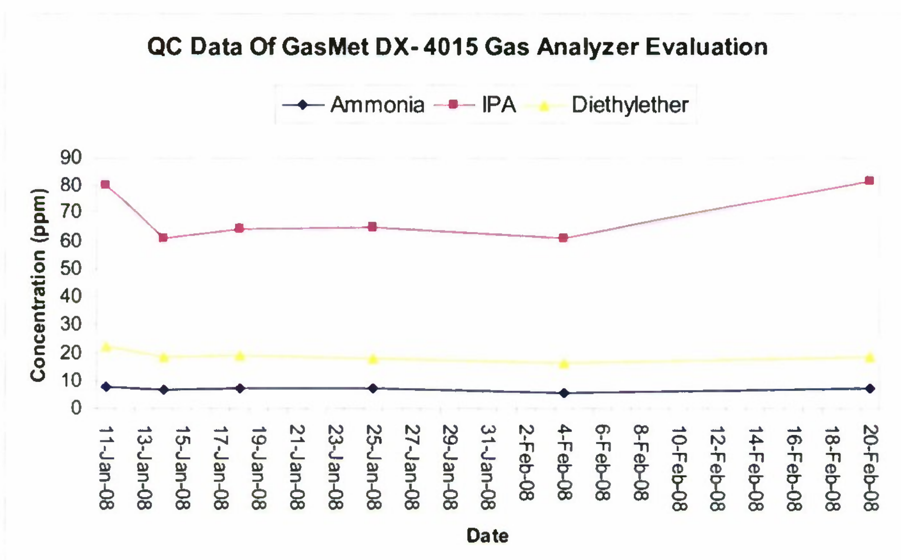

Figure 5. Instrument Performance Verification Data with Ammonia, IPA, and Diethyl Ether

3.2 Procedures.

Validation testing consisted of a multi-tiered process. Tier 1 consisted of a pre- and post-ruggedness LOD estimation for limited analytes that are routinely used to verify DX-4015 FTIR system performance. Instruments were fastened to shaker tables for ruggedness testing. If the instrument showed a marked reduction in performance capability following the ruggedness testing, a determination was made at that time whether to continue testing the instrument. Tier 2 testing was comprised of LOD, dynamic range, inter-operator variability, specificity, and selectivity testing for the CWA and TIC/TIM compounds listed in Tables 1 and 2.

3.3 Tier 1 Testing.

Isopropyl Alcohol, diethyl ether and Ammonia in nitrogen (Table 2) were employed as target analytes for Tier 1 testing. These target analytes are liquids at room temperature and a small amount of each liquid (1 to 10 μL) was injected into a 3 L Tedlar bag. The bag was then filled with nitrogen. Nitrogen was used because of its inertness and because it does not interfere with FTIR analysis of the target analytes used to verify FTIR system performance and sample analysis testing. Gas dilutions were performed (3 to 5 replicates at a low range concentration) of Ammonia, Diethyl ether, and Isopropyl alcohol spiked into Tedlar bags (Figures 5 and 6 and Table 2). The dilutions were used to determine estimated LODs both prior to and following a shaker-table ruggedness test. These estimated LODs were established at the concentration at which the FTIR detector identifies each target analyte in all five replicates.

During ruggedness testing, the instrument was fastened to shaker tables using the same methods used in the ALS Increment 1 mobile laboratories. Ruggedness testing was performed by the ECBC Environmental & Field Test Group IAW MIL-STD-810F, Method 514.5, Procedure I, and Category 4 with the common carrier transportation profile found on pages 24 thru 33 for 3 hr in each of three perpendicular axes. This profile represents normal over-the-road operations (conditions that comprise 90% of the proposed operating environment) equivalent to 1,000 miles. Any instrument for which post-ruggedness LODs increased by ≥100 fold as compared to pre-ruggedness LODs was to be subjected to further testing (see Appendix B).

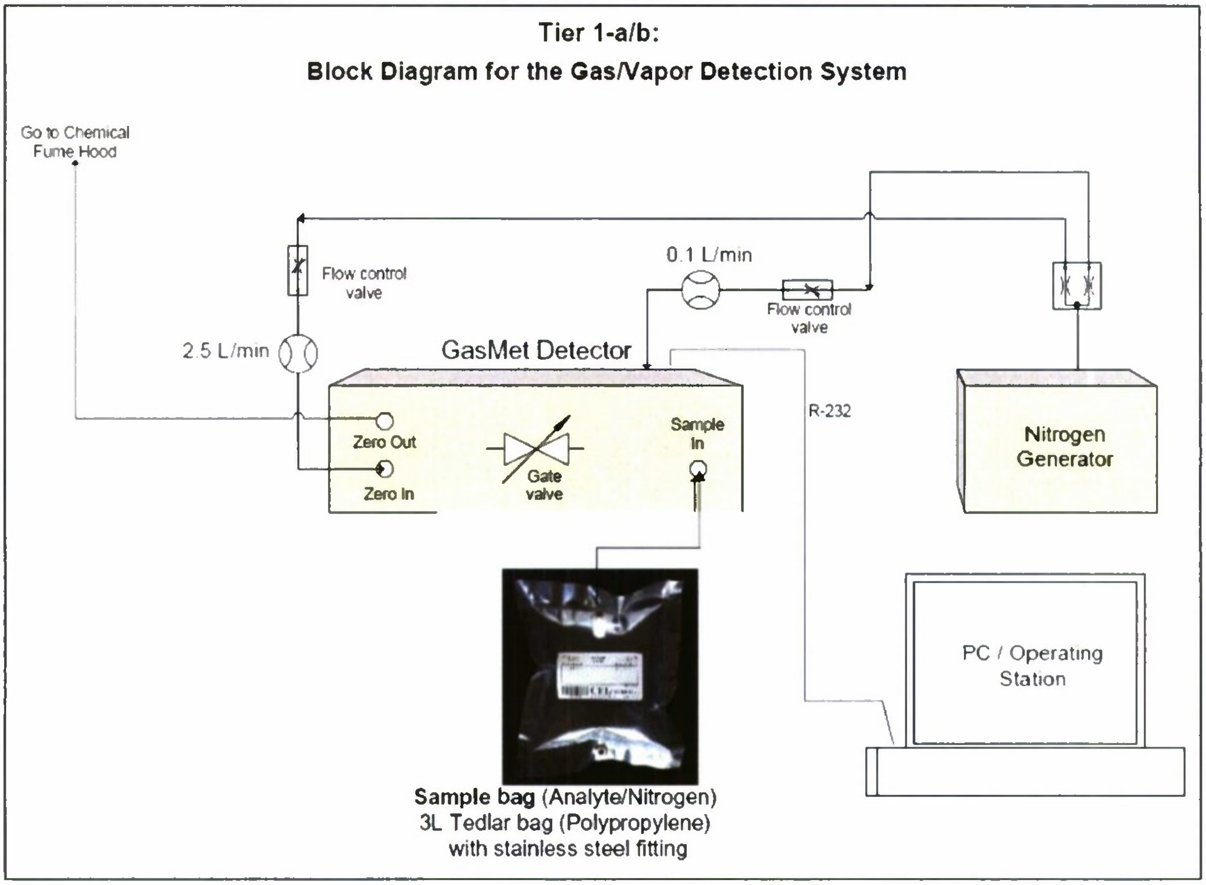

Figure 6. Tier 1 Testing Setup for Pre- and Post-Ruggedness Testing

3.4 Tier 2 Testing.

Testing consisted of LOD, selectivity and specificity testing with the CWA and TIC/TIM analytes listed in Tables 1 and 2. Testing was performed at ECBC, in Building E5830, in a portable chemical hood and/or glovebox (Figure 7). Initially, a gas standard of Diethyl ether, Isopropyl alcohol and Ammonia in a Tedlar bag were analyzed in triplicate to verify sufficient instrument sensitivity to allow testing to continue. The spectral identification match for all replicates was recorded.

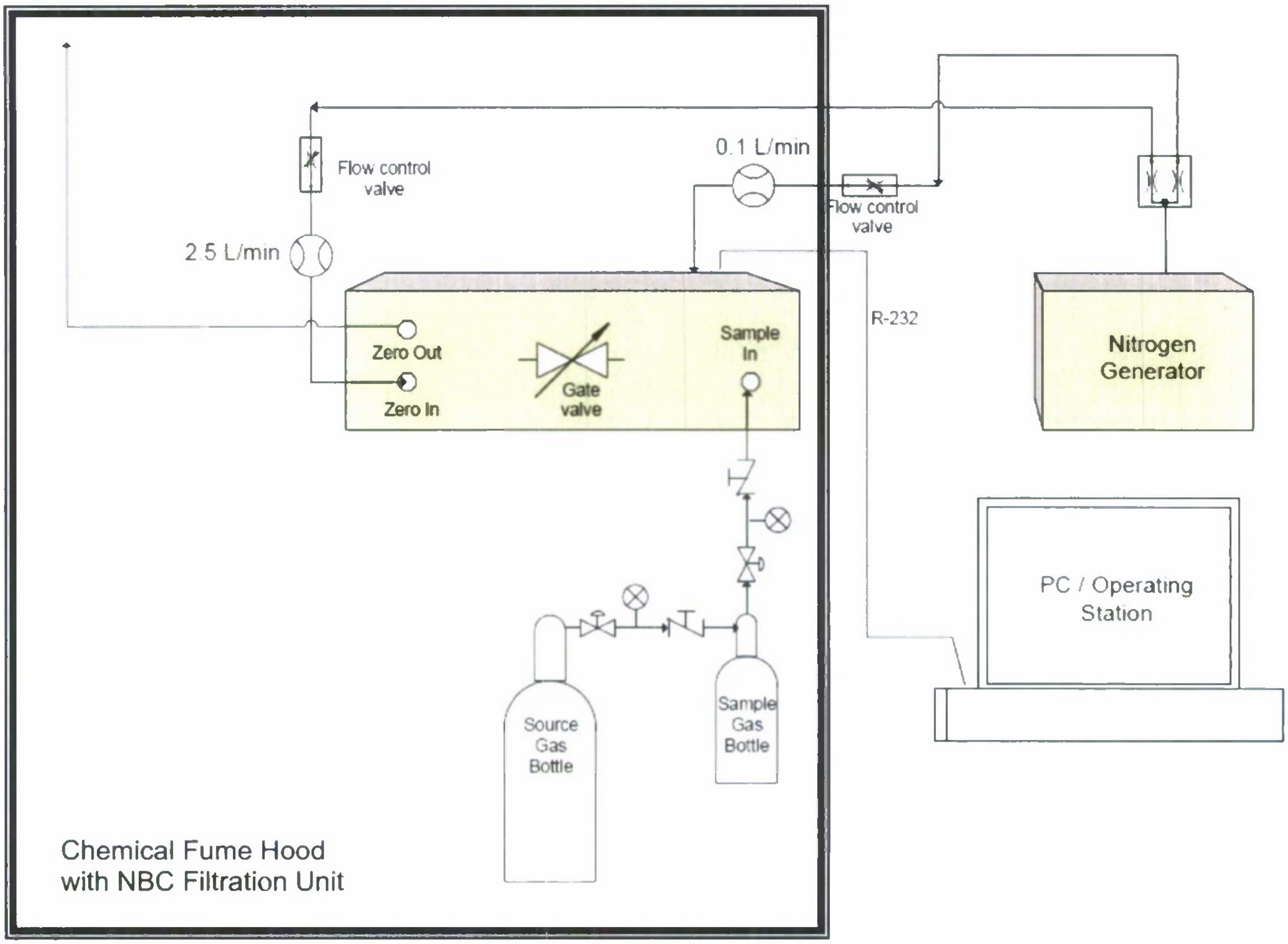

Figure 7. Tier 2 Tcsting Setup

Following the initial verification with Diethyl ether, Isopropyl alcohol, and Ammonia gas standards, the estimated LODs for each analyte were determined by analyzing each single analyte gas standard at the establishcd LOD 22 to 25 times (Table 2). The actual number of analysis ranged from 30 to 60 analytical runs. This was accomplished for each gas by the ease of use of the instrument and its ability to do short timed and accurate analysis from each gas cylinder. The spectral library identification for the target analyte was reported for each replicate concentration, then that concentration was repeated.

Once an LOD was established on the instrument, the analysts performed a dynamic range experiment at concentrations ranging from the established LOD to three to four additional gas analyses above the initial cstimated LODs in five runs each to determine thc LODs and the dynamic range of the instrument. Fivc replicates were analyzed at each concentration during each run. Each analyst used the same sample gas standards for each analyte. Limits of detection were determined based on the following conditions: 22/22 replicates must produce a spectral match of ≥90% for the target analyte. The DX-4015 docs not report identifications as a function of similarity matches or in a quantity for spectral matchcs, but as simple gas compound identification.

Replicates of Phosgene and Hydrogen Cyanide were performed by summa canister to verify that the summa canisters can be used as a sample collection tool for this instrument and to show reproducibility. The ethylene oxide cylinders where emptied during Tier 2 testing. The canisters used were 400 mL summa canisters by volume. When pumped down to a vacuum of approximately 25 mm of Hg a concentration 3 to 5 times less than the actual concentration was recorded. Therefore, it was determined that a larger size of summa canister was needed to accurately measure the concentration of a gas in the 1 M cell at the time of analysis and due to the force of the pump. It was also determined quantitatively that a 1 to 3 L summa canister would increase the accuracy of the analysis in relation to the concentration. However, qualitatively, the DX-4015 identified each gas accurately. Therefore, 22/22 replicates were not performed because each concentration varied over a short period of time when using the 400 mL summa canister.

4. CONCLUSIONS

The testing of the DX-4015 indicated that the instrument was shown to be very specific for the TIC/TIM gases selected for testing (see Tables 4 and 5). The instrument was accurate when measuring each gas based on the concentration of each gas mixture (Figures 7-16) and it showed excellent sensitivity and selectivity for each gas at ambient temperatures. The actual limit of detection (LOD) for each gas selected and tested was shown to be less than 1.0 parts per million (ppm). An actual LOD study was not performed because the capability of the instrument to identify compounds below 1.0 ppm was not determined until the time of analysis with the use of predetermined concentrations that had already been purchased and due to project timelines. In the Tier 2 Testing Setup, the analyzer and gas bottles were placed in a chemical fume hood equipped with a NBC filtration unit.

Any further analysis would have involved more time and funding, which was not feasible. It must be pointed out that during the initial testing of the DX-4015 instruments; one instrument was shaken in accordance with military standards and returned for verification and performance checks. Initially, the instrument functioned correctly but failed on start up 2 weeks later. The instrument was shipped back to the manufacturer who determined that the power supply had dislodged and shorted out the laser. The manufacturer was aware of the possibility of this failure and fixed both DX-4015 instruments at no cost.

A second shake test was performed followed by verification and performance testing. The instrument continued to work, indicating that the manufacturer had indeed fixed the problem. All newly purchased or subsequently purchased DX -4015 instruments will include this equipment upgrade. A 90 to 95% confidence interval was easily achieved because the instrument was able to acquire data points continuously within a very short time frame or by shortening the data collection time. All data points for this test and for each gas tested were collected at 20 s intervals. Also, testing of the summa canisters using the internal DX-4015 pump indicated that because of the size of the canister (400 mL), there was not enough sample gas collected to completely fill the gas cell of the instrument during analysis and accurately measure the concentration. However, the instrument identified each target gas, but the actual concentration of each gas reported had to be multiplied by a factor of 4 to 6 for a more accurate measurement. It was also determined during testing that in order to achieve an accurate concentration of gas using summa canisters, the canisters must be 1 to 3 L or larger.

The data were collected and provided to PM-CM to be used for the instrument's performance verification and to identify gaseous CWA/TIC/TIM compounds for use in the National Guard Bureau's Increment I Analytical Laboratory Systems.

Table 4. Tier 1 Testing Results Summary*

Test	Date	Time	SpectrumFile	LibraryFile	Chemical	[Measure]	Unit	Mean	Stdev
Tier 1a	10/16/2007	17:16:14	C:\CALCMETSAMPLES\ALS\20071016\IPA_5uL_00099.SPE	DX071236ALIB	IPA	106.89	ppm	114.52	10.34
	10/16/2007	17:26:15	C:\CALCMETSAMPLES\ALS\20071016\IPA_5uL_2nd_00101.SPE	DX071236ALIB		111.49	ppm		
	10/16/2007	17:26:36	C:\CALCMETSAMPLES\ALS\20071016\IPA_5uL_2nd_00102.SPE	DX071236ALIB		124.66	ppm		
	10/16/2007	17:26:57	C:\CALCMETSAMPLES\ALS\20071016\IPA_5uL_2nd_00103.SPE	DX071236ALIB		126.09	ppm		
	10/16/2007	17:29:19	C:\CALCMETSAMPLES\ALS\20071016\IPA_5uL_2nd_00104.SPE	DX071236ALIB		103.44	ppm		
	10/24/2007	17:41:55	C:\CALCMETSAMPLES\ALS\20071024\AMM_5uL_00010.SPE	DX071236ALIB	Ammonia	25.85	ppm	27.35	1.30
	10/24/2007	17:42:16	C:\CALCMETSAMPLES\ALS\20071024\AMM_5uL_00011.SPE	DX071236ALIB		28.03	ppm		
	10/24/2007	17:42:37	C:\CALCMETSAMPLES\ALS\20071024\AMM_5uL_00012.SPE	DX071236ALIB		28.17	ppm		
	10/24/2007	17:48:31	C:\CALCMETSAMPLES\ALS\20071024\DEE_1uL_00014.SPE	DX071236ALIB	Diethylether	59.86	ppm	64.45	3.68
	10/24/2007	17:48:52	C:\CALCMETSAMPLES\ALS\20071024\DEE_1uL_00015.SPE	DX071236ALIB		62.54	ppm		
	10/24/2007	17:49:13	C:\CALCMETSAMPLES\ALS\20071024\DEE_1uL_00016.SPE	DX071236ALIB		62.31	ppm		
	10/24/2007	18:05:24	C:\CALCMETSAMPLES\ALS\20071024\DEE_1uL_2_00022.SPE	DX071236ALIB		64.35	ppm		
	10/24/2007	18:05:45	C:\CALCMETSAMPLES\ALS\20071024\DEE_1uL_2_00023.SPE	DX071236ALIB		68.73	ppm		
	10/24/2007	18:06:06	C:\CALCMETSAMPLES\ALS\20071024\DEE_1uL_2_00024.SPE	DX071236ALIB		68.92	ppm		
Tier 1b	11/14/2007	9:58:18	C:\CALCMETSAMPLES\ALS\20071114\IPA_5uL_1_00001.SPE	DX071236ALIB	IPA	94.02	ppm	136.21	36.66
	11/14/2007	9:58:39	C:\CALCMETSAMPLES\ALS\20071114\IPA_5uL_1_00002.SPE	DX071236ALIB		97.76	ppm		
	11/14/2007	10:23:04	C:\CALCMETSAMPLES\ALS\20071114\IPA_5uL_2_00010.SPE	DX071236ALIB		166.59	ppm		
	11/14/2007	10:23:25	C:\CALCMETSAMPLES\ALS\20071114\IPA_5uL_2_00011.SPE	DX071236ALIB		176.37	ppm		
	11/14/2007	10:23:46	C:\CALCMETSAMPLES\ALS\20071114\IPA_5uL_2_00012.SPE	DX071236ALIB		144.32	ppm		
	11/14/2007	10:31:40	C:\CALCMETSAMPLES\ALS\20071114\AMM_5uL_1_00014.SPE	DX071236ALIB	Ammonia	39.01	ppm	37.00	4.27
	11/14/2007	10:32:01	C:\CALCMETSAMPLES\ALS\20071114\AMM_5uL_1_00015.SPE	DX071236ALIB		41.62	ppm		
	11/14/2007	10:32:22	C:\CALCMETSAMPLES\ALS\20071114\AMM_5uL_1_00016.SPE	DX071236ALIB		41.64	ppm		
	11/14/2007	10:37:28	C:\CALCMETSAMPLES\ALS\20071114\AMM_5uL_2_00018.SPE	DX071236ALIB		32.99	ppm		
	11/14/2007	10:37:49	C:\CALCMETSAMPLES\ALS\20071114\AMM_5uL_2_00019.SPE	DX071236ALIB		34.19	ppm		
	11/14/2007	10:38:10	C:\CALCMETSAMPLES\ALS\20071114\AMM_5uL_2_00020.SPE	DX071236ALIB		32.52	ppm		
	11/14/2007	10:47:22	C:\CALCMETSAMPLES\ALS\20071114\DEE_1uL_1_00024.SPE	DX071236ALIB	Diethylether	63.16	ppm	66.23	2.43
	11/14/2007	10:47:43	C:\CALCMETSAMPLES\ALS\20071114\DEE_1uL_1_00025.SPE	DX071236ALIB		66.62	ppm		
	11/14/2007	10:48:04	C:\CALCMETSAMPLES\ALS\20071114\DEE_1uL_1_00026.SPE	DX071236ALIB		66.31	ppm		
	11/14/2007	10:53:06	C:\CALCMETSAMPLES\ALS\20071114\DEE_1uL_2_00028.SPE	DX071236ALIB		63.96	ppm		
	11/14/2007	10:53:27	C:\CALCMETSAMPLES\ALS\20071114\DEE_1uL_2_00029.SPE	DX071236ALIB		67.94	ppm		
	11/14/2007	10:53:48	C:\CALCMETSAMPLES\ALS\20071114\DEE_1uL_2_00030.SPE	DX071236ALIB		65.16	ppm		

*Note: When the Gasmet™ Calcmet software reports a negative value, one of two things occurs. If the value is within ~3% of the lowest reference concentration, then this is (absolute value) total measurement noise. If the value is negative by more than the (absolute value) ~3% reference value, the Gasmet™ Calcmet software is overcompensating for some other compound (reference spectra) in the interference table and the analysis settings may need to be optimized.

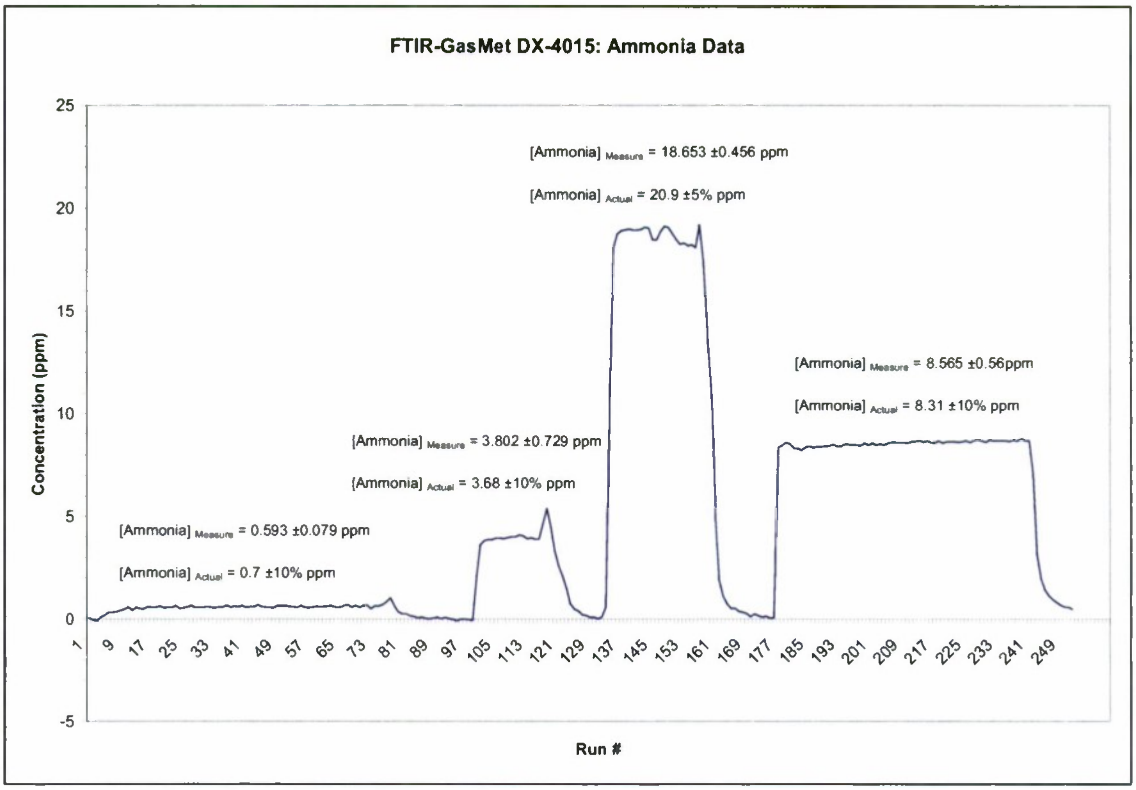

Figure 8. Ammonia Data from Tier 2 Test at Four Different Concentrations

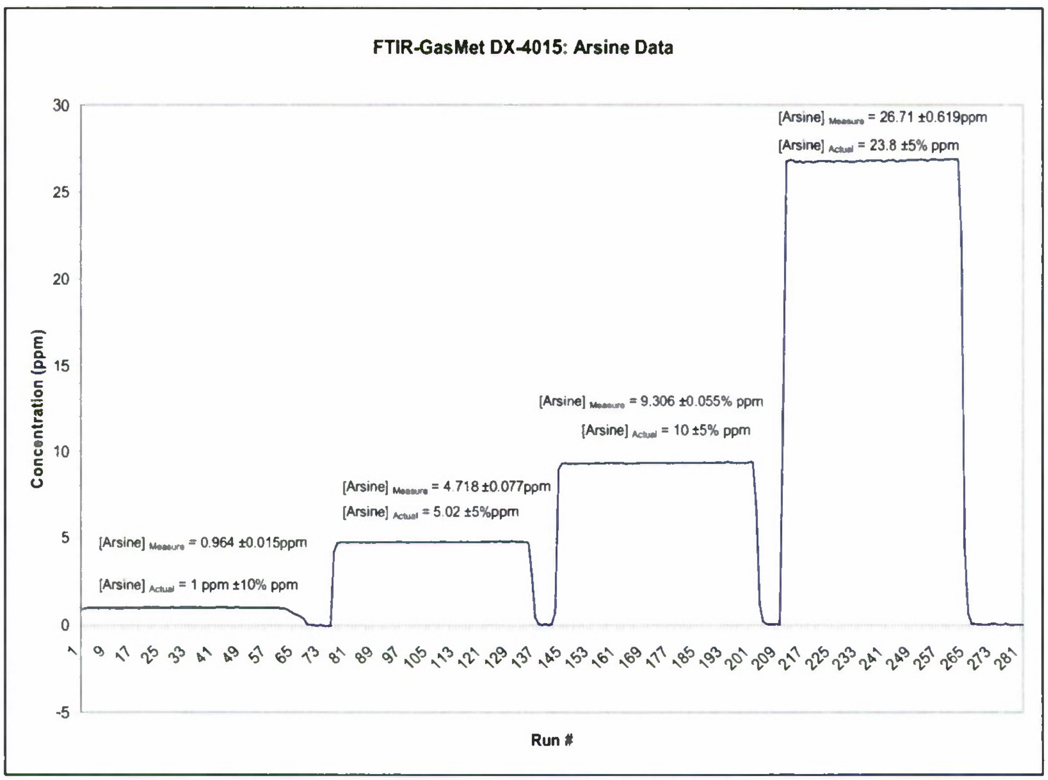

Figure 9. Arsine Data from Tier 2 Test at Four Different Concentrations

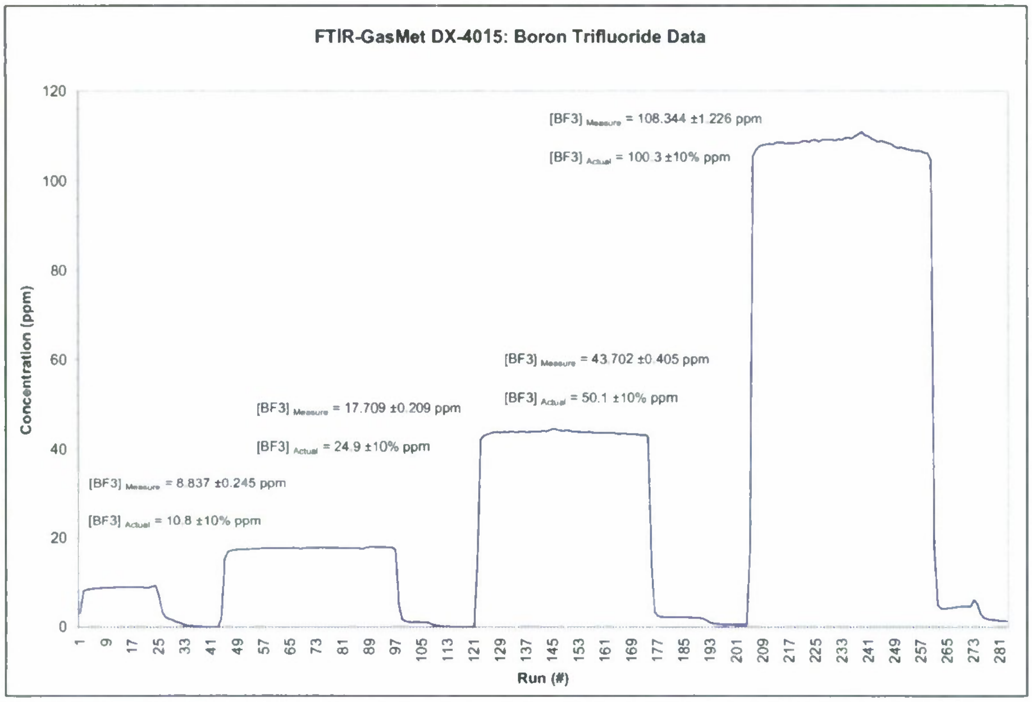

Figure 10. Boron Trifluoride Data from Tier 2 Test at Four Different Concentrations

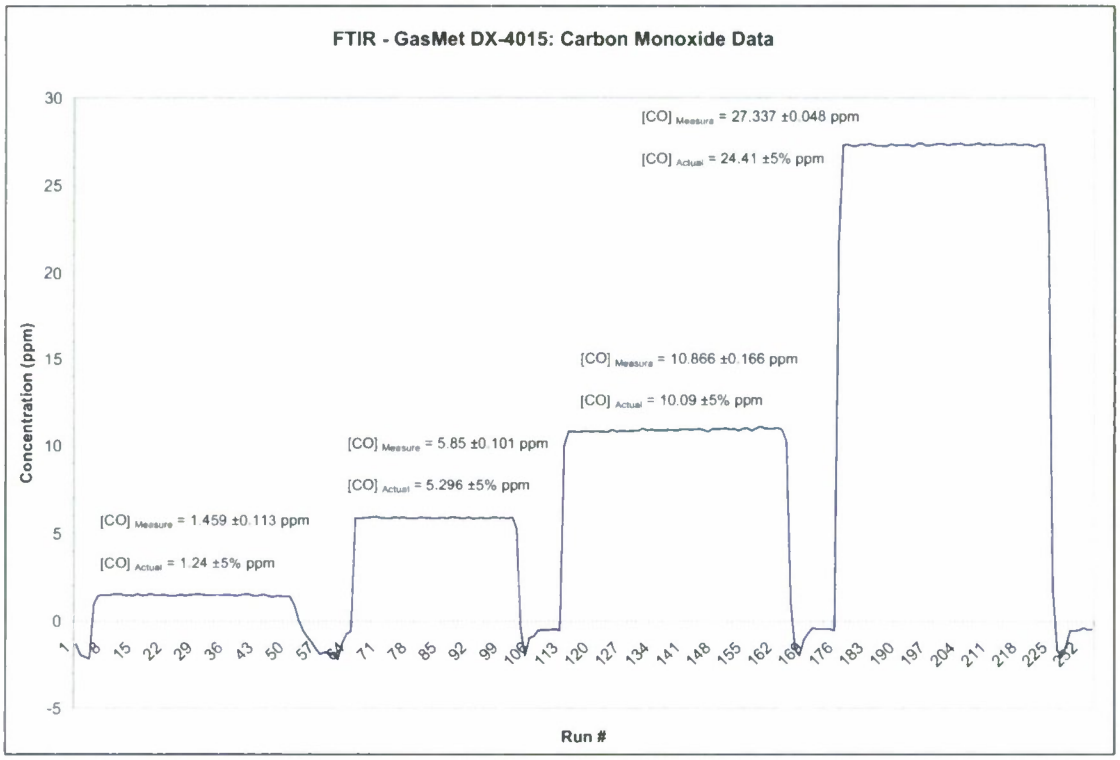

Figure 11. Carbon Monoxide Data at Four Different Concentrations from Tier 2 Test

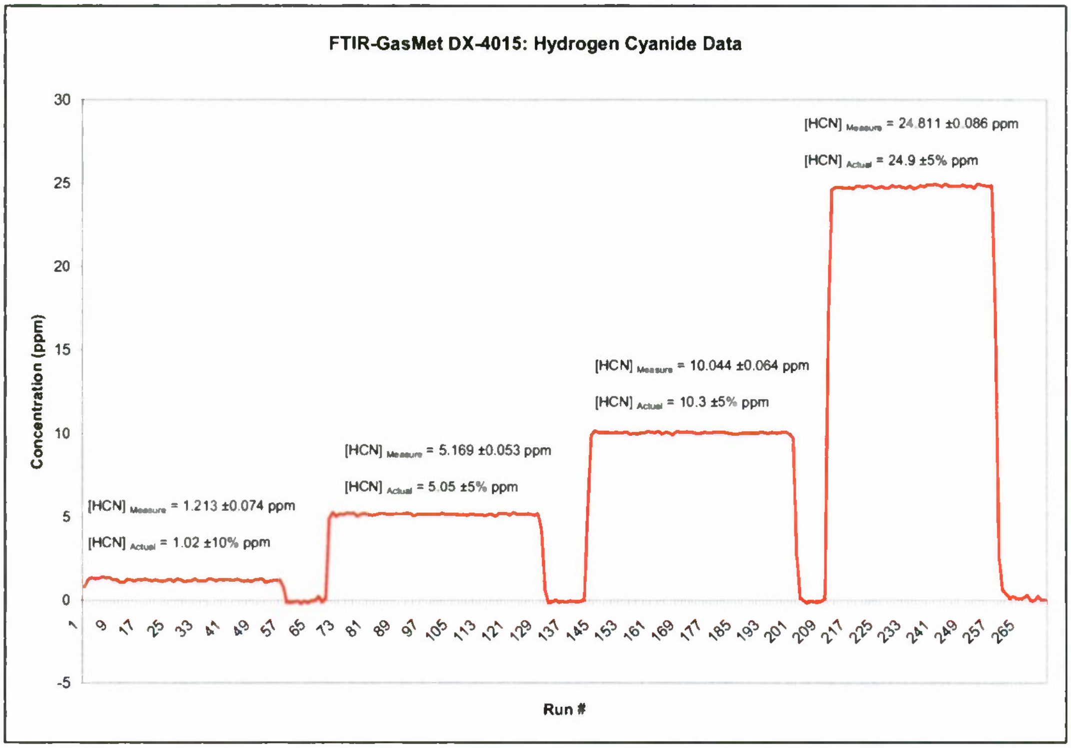

Figure 12. Hydrogen Cyanide Data at Four Different Concentrations from Tier 2 Test

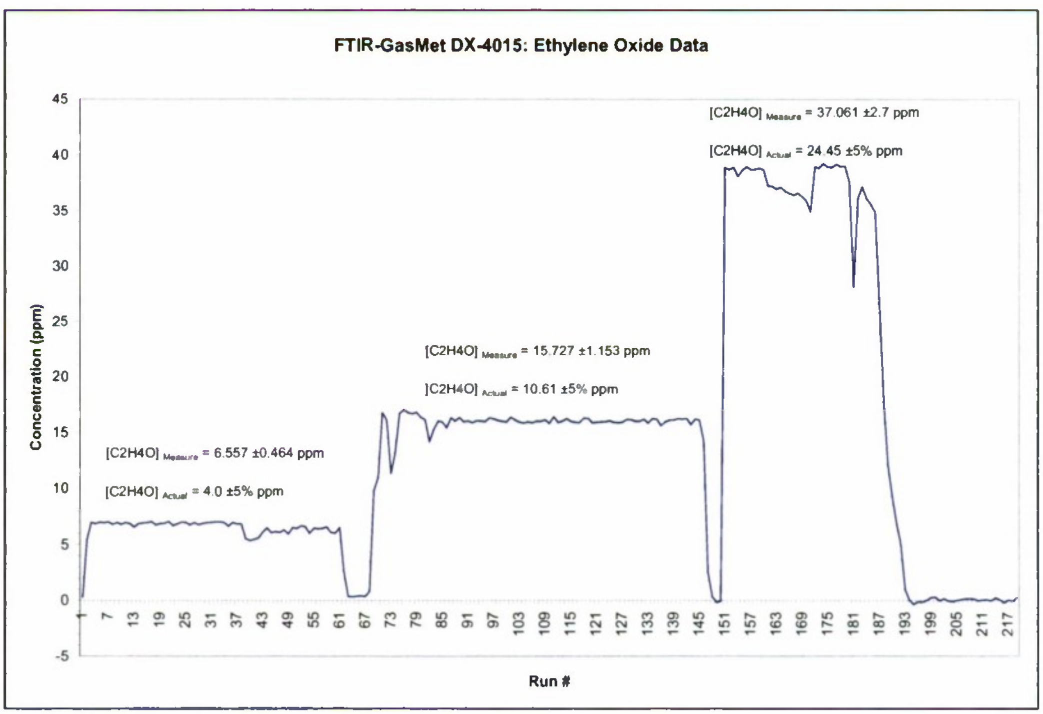

Figure 13. Ethylene Oxide Data at Three Different Concentrations from Tier 2 Test

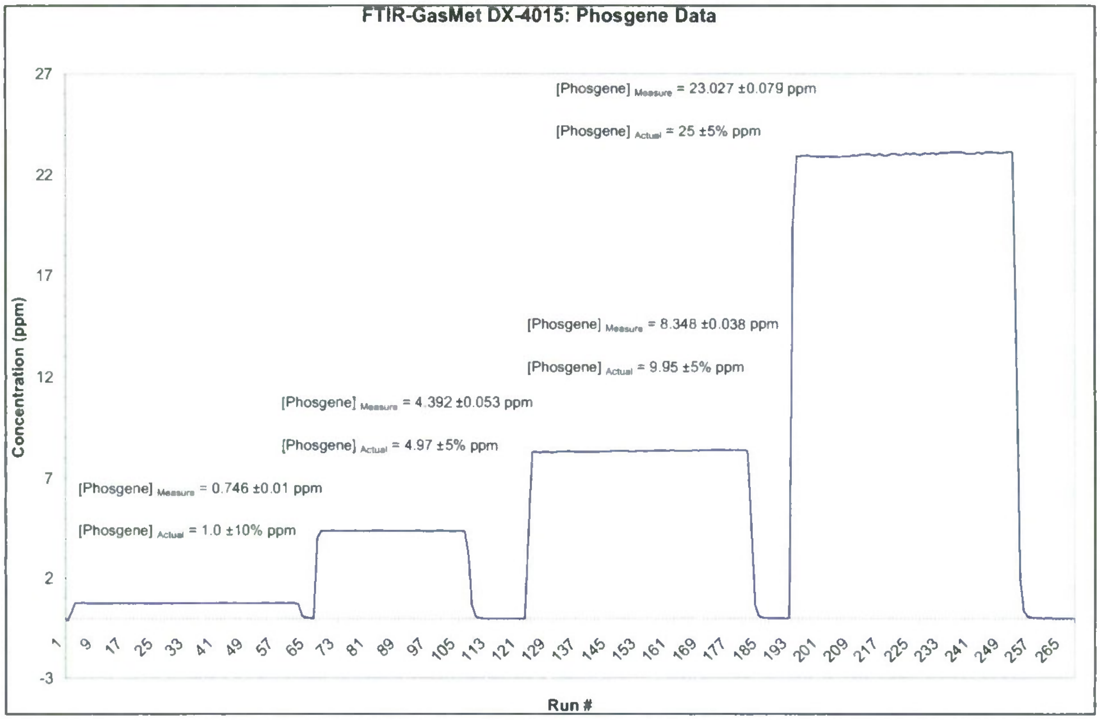

Figure 14. Phosgene Data at Four Different Concentrations from Tier 2 Test

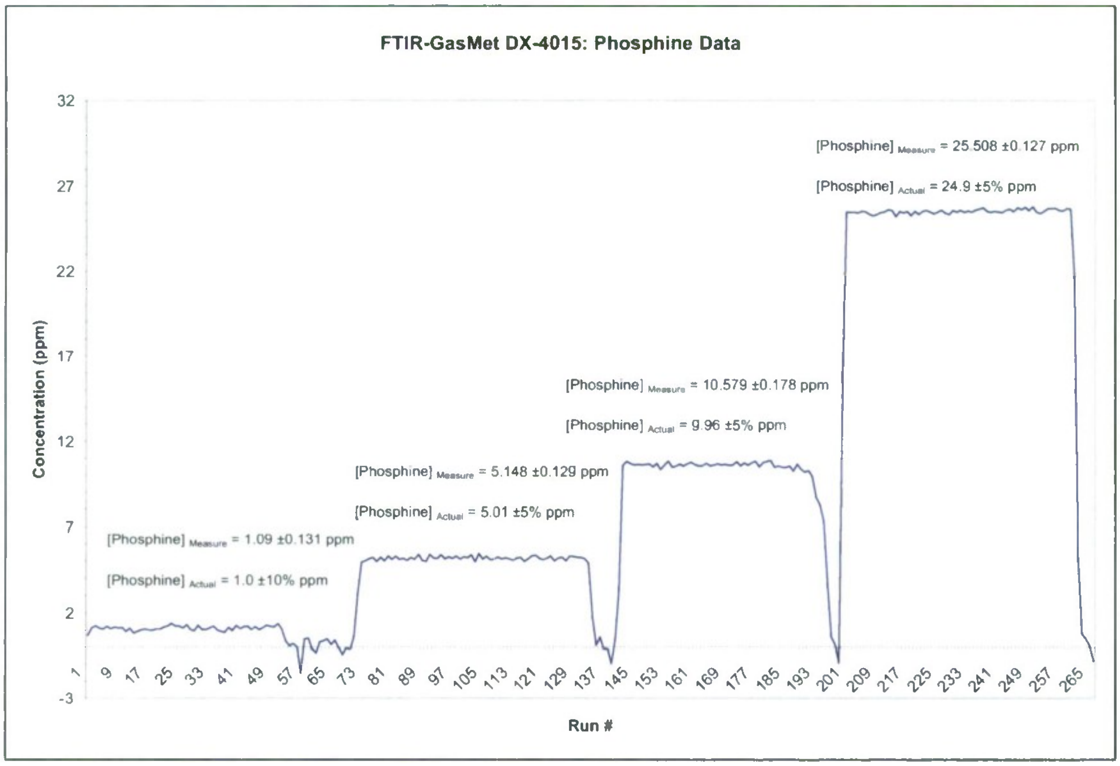

Figure 15. Phosphine Data at Four Different Concentrations from Tier 2 Test

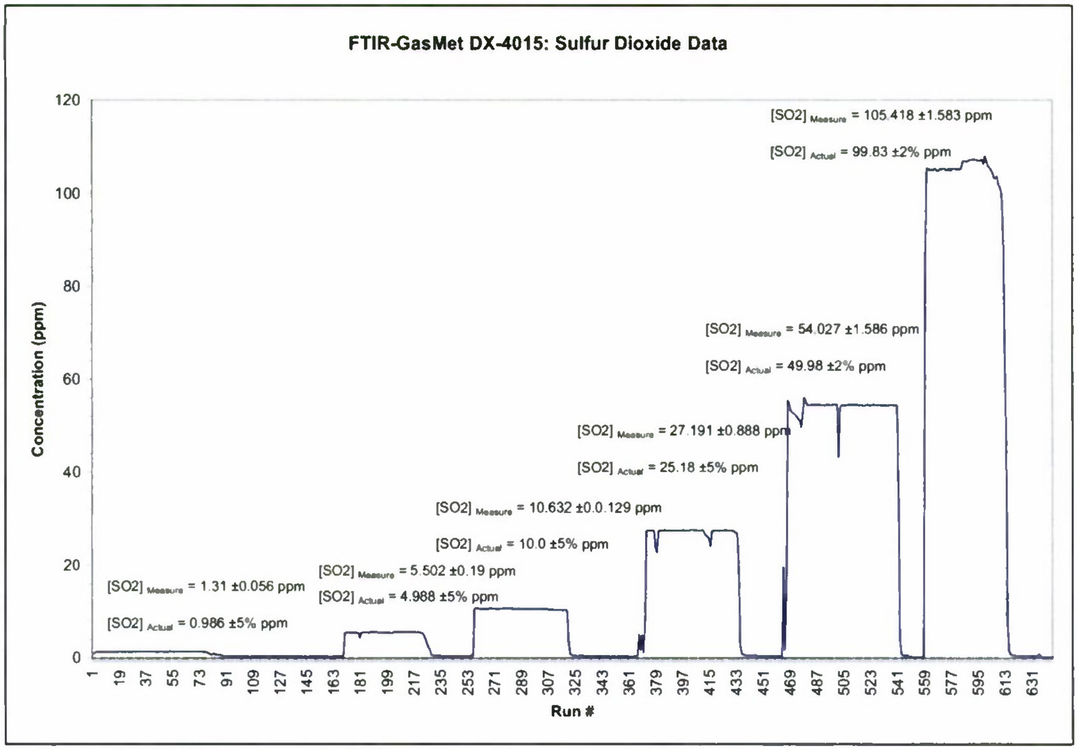

Figure 16. Sulfur Dioxide Data at Five Different Concentrations from Tier 2 Test

Table 5. Tier 2 Testing Results Summary*

Testing Gas	Standard Analytical Certification No.	[Standard]$_{Actual}$ (ppm)	FTIR-GasMet DX-4015 Reading						
			Mean	Delta	Sigma	±CI (95%)	±CI (90%)	C.R.(95%)	C.R.(90%)
Ammonia	82-124118422-1	0.7 ±10%	0.593	0.62	0.079	0.019	0.016	0.593 ±0.019	0.593 ±0.016
	82-124118423-1	3.88 ±10%	3.602	3.31	0.729	0.323	0.267	3.602 ±0.323	3.602 ±0.267
		6.31 ±10%	6.565	0.56	0.123	0.035	0.031	6.565 ±0.035	6.565 ±0.031
	82-124118425-1	20.9 ±5%	16.653	1.64	0.456	0.197	0.163	16.653 ±0.197	16.653 ±0.163
Arsine	FF49303	1.00 ±10%	0.964	0.1	0.015	0.004	0.003	0.964 ±0.004	0.964 ±0.003
	FF51170	5.02 ±5%	4.716	0.61	0.077	0.020	0.017	4.716 ±0.02	4.716 ±0.017
	FF51156	10.00 ±5%	9.306	0.42	0.055	0.014	0.012	9.306 ±0.014	9.306 ±0.012
	FF49331	23.8 ±5%	26.710	4.59	0.619	0.172	0.144	26.71 ±0.172	26.71 ±0.144
Boron Trifluoride	XF0039268	10.6 ±10%	8.637	1.19	0.245	0.109	0.090	8.637 ±0.109	8.637 ±0.09
	XF0002126	24.9 ±10%	17.709	1.07	0.209	0.059	0.049	17.709 ±0.059	17.709 ±0.049
	XF0029268	50.1 ±10%	43.702	2.43	0.405	0.114	0.095	43.702 ±0.114	43.702 ±0.095
	XF0016746	100.3 ±10%	108.344	8.41	1.226	0.335	0.279	108.344 ±0.335	108.344 ±0.279
Carbon Monoxide	82-124118914-1	1.24 ±5%	1.459	0.6	0.113	0.034	0.028	1.459 ±0.034	1.459 ±0.028
	82-124118916-1	5.298 ±5%	5.850	0.65	0.101	0.034	0.028	5.85 ±0.034	5.85 ±0.028
	82-124118919-1	10.09 ±5%	10.866	1.09	0.166	0.047	0.039	10.866 ±0.047	10.866 ±0.039
	82-124118922-1	24.41 ±5%	27.337	0.19	0.046	0.014	0.012	27.337 ±0.014	27.337 ±0.012
Hydrogen Cyanide	FF51150	1.02 ±10%	1.213	0.34	0.074	0.020	0.017	1.213 ±0.02	1.213 ±0.017
	FF49264	5.05 ±5%	5.169	0.25	0.053	0.014	0.012	5.169 ±0.014	5.169 ±0.012
	FF49200	10.3 ±5%	10.044	0.4	0.064	0.017	0.014	10.044 ±0.017	10.044 ±0.014
	FF51171	24.9 ±5%	24.811	0.36	0.066	0.026	0.022	24.811 ±0.026	24.811 ±0.022
Ethylene Oxide	63-124118839-2	4.0 ±5%	6.557	1.7	0.464	0.121	0.101	6.557 ±0.121	6.557 ±0.101
	63-124118839-3	10.81 ±5%	15.727	7.2	1.153	0.262	0.219	15.727 ±0.262	15.727 ±0.219
	63-124118839-1	24.45 ±5%	37.061	12.61	2.700	0.913	0.760	37.061 ±0.913	37.061 ±0.760
Phosgene	FF50014	1.0 ±10%	0.746	0.08	0.010	0.003	0.002	0.746 ±0.003	0.746 ±0.002
	FF49196	4.97 ±5%	4.392	0.34	0.053	0.017	0.014	4.392 ±0.017	4.392 ±0.014
	FF49271	9.95 ±5%	8.348	0.14	0.036	0.010	0.008	8.348 ±0.01	8.348 ±0.008
	FF49202	25 ±5%	23.027	0.24	0.079	0.021	0.018	23.027 ±0.021	23.027 ±0.016
Phosphine	FF51174	1.0 ±10%	1.090	0.58	0.131	0.037	0.031	1.09 ±0.037	1.09 ±0.031
	FF49240	5.01 ±5%	5.146	0.57	0.129	0.033	0.028	5.146 ±0.033	5.146 ±0.028
	FF51154	9.98 ±5%	10.579	0.97	0.178	0.051	0.042	10.579 ±0.051	10.579 ±0.042
	FF49249	24.9 ±5%	25.506	0.6	0.127	0.033	0.028	25.506 ±0.033	25.506 ±0.028
Sulfur Dioxide	82-124118906-1	0.986 ±5%	1.310	0.39	0.056	0.013	0.011	1.31 ±0.013	1.31 ±0.011
	82-124118907-1	4.988 ±5%	5.502	1.35	0.190	0.052	0.044	5.502 ±0.052	5.502 ±0.044
		10.0 ±5%	10.632	0.68	0.129	0.033	0.027	10.632 ±0.033	10.632 ±0.027
	82-124118928-1	25.16 ±5%	27.191	4.81	0.886	0.227	0.190	27.191 ±0.227	27.191 ±0.19
	82-124118902-1	49.98 ±2%	54.027	12.8	1.586	0.370	0.309	54.027 ±0.37	54.027 ±0.309
	82-124118904-1	99.83 ±2%	105.418	8.24	1.563	0.450	0.375	105.418 ±0.45	105.418 ±0.375

*Note: The CI (Confidence Interval) is used to indicate the reliability of an estimate and an indication of the accuracy with which a parameter is known using the estimate. Therefore a 95% confidence interval is an interval generated by a process that is right 95% of the time. Similarly, a 90% confidence interval is an interval generated by a process that is right 90% of the time.

The CR (Confidence Range) is the mean value of a population or set of data within plus or minus of the defined accuracy of the mean generated by the data set and with which the level of confidence will be defined.

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APPENDIX A
LIMITS OF DETECTION

TICs/TIM (Threshold)	Hazard Class	Limit of Detection	Primary Technology (†)	Derived From:
1. Ammonia (g)	High	250μg/mL	IMS/GCMS	ITF-25
2. Arsine (g)	High	250μg/mL	IMS/GCMS	ITF-25
3. Boron trichloride (g)	High	250μg/mL	IMS/GCMS	ITF-25
4. Boron trifluoride (g)	High	250μg/mL	IMS/GCMS	ITF-25
5. Carbon disulfide	High	250μg/mL	IMS/GCMS	ITF-25
6. Chlorine (g)	High	250μg/mL	IMS/GCMS	ITF-25
7. Diborane (g)	High	250μg/mL	IMS/GCMS	ITF-25
8. Ethylene oxide (g)	High	250μg/mL	IMS/GCMS	ITF-25
9. Fluorine (g)	High	250μg/mL	IMS/GCMS	ITF-25
10. Formaldehyde	High	250μg/mL	IMS/GCMS	ITF-25
11. Hydrogen bromide (g)	High	250μg/mL	IMS/GCMS	ITF-25
12. Hydrogen chloride	High	Neat	IMS/FTIR	ITF-25
13. Hydrogen cyanide (g)	High	250μg/mL	IMS/GCMS	ITF-25
14. Hydrogen fluoride (g)	High	Neat	IMS/FTIR	ITF-25
15. Hydrogen sulfide (g)	High	250μg/mL	IMS/GCMS	ITF-25
16. Nitric Acid, fuming	High	Neat	IMS/FTIR	ITF-25
17. Phosgene (g)	High	250μg/mL	IMS/GCMS	ITF-25
18. Phosphorus trichloride	High	250μg/mL	GCMS/FTIR	ITF-25
19. Sulfur dioxide (g)	High	250μg/mL	IMS/GCMS	ITF-25
20. Tungsten hexafluoride (g)	High	250μg/mL	IMS/GCMS	ITF-25
21. Sulfuric Acid	High	Neat	IMS/FTIR	ITF-25
22. Acrolein	Medium	250μg/mL	IMS/GCMS	ITF-25
23. Acetone Cyanohydrin	Medium	250μg/mL	IMS/GCMS	ITF-25
24. Acrylonitrile	Medium	250μg/mL	IMS/GCMS	ITF-25
25. Ally Alcohol	Medium	100μg/mL	IMS/GCMS	ITF-25
26. Allylamine	Medium	100μg/mL	IMS/GCMS	ITF-25
27. Allyl chlorocarbonate	Medium	250μg/mL	IMS/GCMS	ITF-25
28. Boron tribromide	Medium	250μg/mL	IMS/GCMS	ITF-25
29. Carbon monoxide (g)	Medium	250μg/mL	IMS/GCMS	ITF-25
30. Carbonyl sulfide (g)	Medium	250μg/mL	IMS/GCMS	ITF-25
31. Chloroacetone	Medium	100μg/mL	IMS/GCMS	ITF-25
32. Chloroacetonitrile	Medium	100μg/mL	IMS/GCMS	ITF-25
33. Chlorosulfonic acid	Medium	Neat	IMS/FTIR	ITF-25
34. Diketene	Medium	250μg/mL	IMS/GCMS	ITF-25
35. 1,2-Dimethylhydrazine	Medium	250μg/mL	IMS/GCMS	ITF-25
36. Ethylene dibromide	Medium	250μg/mL	IMS/GCMS	ITF-25
37. Hydrogen Selenide (g)	Medium	250μg/mL	IMS/GCMS	ITF-25
38. Methanesulfonyl chloride	Medium	250μg/mL	IMS/GCMS	ITF-25
39. Methyl bromide (g)	Medium	250μg/mL	IMS/GCMS	ITF-25
40. Methyl chloroformate	Medium	250μg/mL	IMS/GCMS	ITF-25
41. Methyl chlorosilane	Medium	250μg/mL	IMS/GCMS	ITF-25
42. Methyl hydrazine	Medium	250μg/mL	IMS/GCMS	ITF-25
43. Methyl isocyanate	Medium	250μg/mL	IMS/GCMS	ITF-25

Requires air sampling techniques
† When multiple technologies are listed, first one is primary
(g) Compound is a gas at room temperature

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APPENDIX B
ENVIRONMENTAL AND FIELD "SHAKER" TESTING REPORT

FOR AND RESULTS OF TESTS	PAGE NO 1	NO. OF PAGES 13

SECTION A - REQUEST FOR TEST

1. TO: (Include ZIP Code)	2. FROM: (Include ZIP Code)
Environmental & Field Test Group Engineering Dir ECBC Bldg. E-3607, Beach Point Rd Gunpowder, MD 21010	Ryan Kuhns and Scott Speed, Mobile Laboratory Team
3. PRIME CONTRACTOR AND ADDRESS (Include ZIP Code) CONTRACTOR NUMBER NA	4. MANUFACTURING PLANT NAME AND ADDRESS (Include ZIP Code) P.O. NUMBER NA

5. END ITEM AND/OR PROJECT	6. SAMPLE NUMBER	7. LOT NO	8. REASON FOR SUBMITTAL	9. DATE SUBMITTED
			Filter performance testing	20071220

10. MATERIAL TO BE TESTED	10a. QUANTITY SUBMITTED	11. QUANTITY REPRESENTED	12. SPEC. & AMEND AND/OR DRAWING NO. & REV FOR SAMPLE & DATE
GASMET Unit	1		

13. PURCHASED FROM OR SOURCE	14. SHIPMENT METHOD	15. DATE SAMPLED AND SUBMITTED BY
NA	Delivered	

16. REMARKS AND/OR SPECIAL INSTRUCTIONS AND/OR WAIVERS

Item to be Secured Vibration tested IAW MIL-STD-810F Common Carrier profile

17. SEND REPORT OF TEST TO

Distribution - 1 hard copy and one electronic copy to be picked up with test sample by Scott Speed

SECTION B - RESULTS OF TEST (Continue on plain white paper if more space is required)

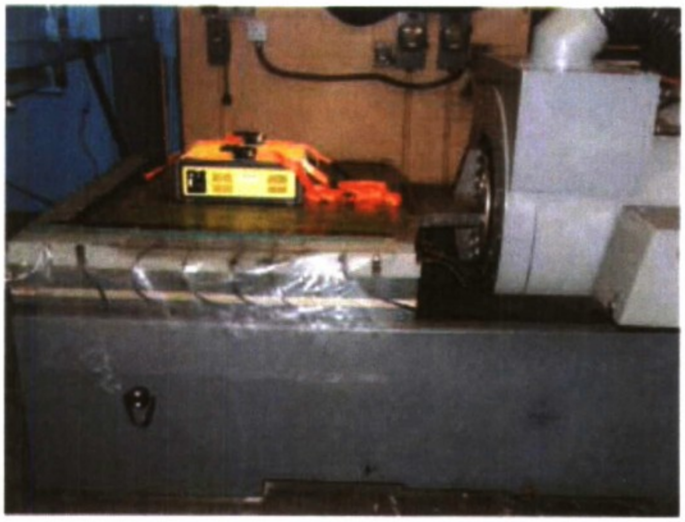
1. DATE SAMPLE RECEIVED	2. DATE RESULTS REPORTED	3. LAB REPORT NUMBER
20071220	20071227	EFT Data Report #O122007-004

TEST PERFORMED RESULTS OF TEST SAMPLE RESULT REQUIREMENTS 4

See EFT Data Report #O122007-004

DATE	TYPED NAME AND TITLE OF PERSON CONDUCTING TEST	SIGNATURE
20071227	Gregory S. Walton / Electronics Technician	

Longitudinal Axis

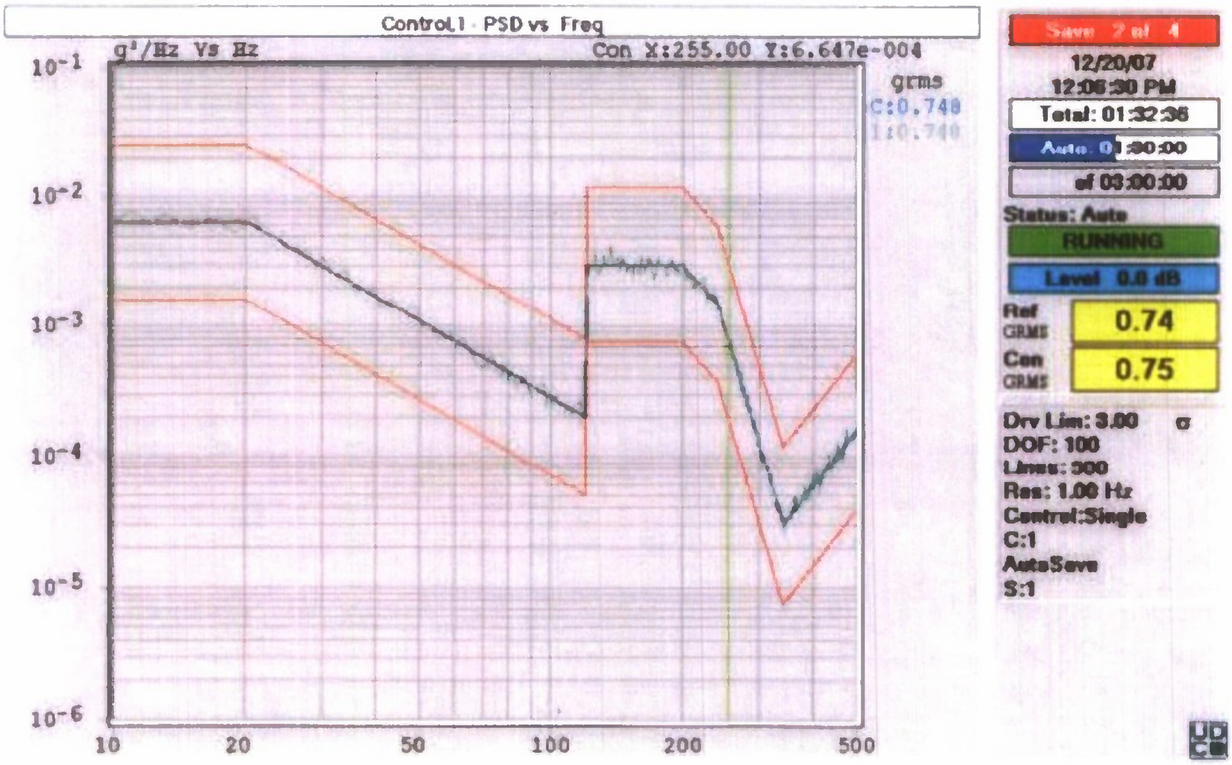


Random Test Report

SETUP NAME: Mil-Std-810F_Long_CC
SETUP DESC: MIL-STD-810F Common Carrier Longitudinal Axis
RUN NAME: **Gasmet Long Dec 07**
USER/PROJECT FOLDER: MIL-STD-810F
SAVE NUMBER: 2

STATUS INFORMATION

TEST EVENT TIME: Thursday, December 20, 2007 at 12:06:30 PM
TEST STATUS: RUNNING
TEST MODE: AUTO
TOTAL TIME ELAPSED (HH:MM:SS): 01:32:36
AUTO TIME ELAPSED (HH:MM:SS): **01:30:00**
TEST LEVEL: 0.0 dB
REFERENCE: 0.74 g rms
CONTROL: 0.75 g rms

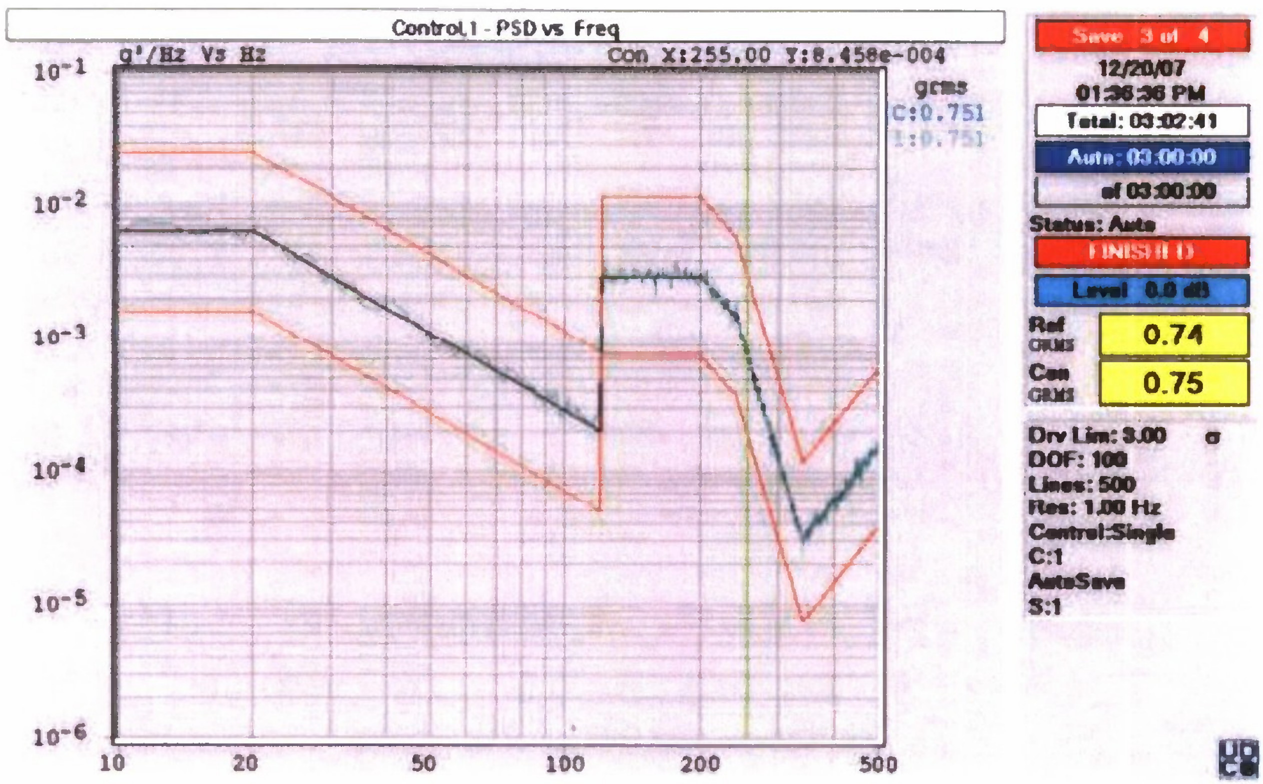


Random Test Report

SETUP NAME: Mil-Std-810f_Long_CC
SETUP DESC: MIL-STD-810F Common Carrier Longitudinal Axis
RUN NAME: **Gasmet Long Dec 07**
USER/PROJECT FOLDER: MIL-STD-810F
SAVE NUMBER: 3

STATUS INFORMATION

TEST EVENT TIME: Thursday, December 20, 2007 at 01:36:36 PM
TEST STATUS: FINISHED
TEST MODE: AUTO
TOTAL TIME ELAPSED (HH:MM:SS): 03:02:41
AUTO TIME ELAPSED (HH:MM:SS): **03:00:00**
TEST LEVEL: 0.0 dB
REFERENCE: 0.74 g rms
CONTROL: 0.75 g rms

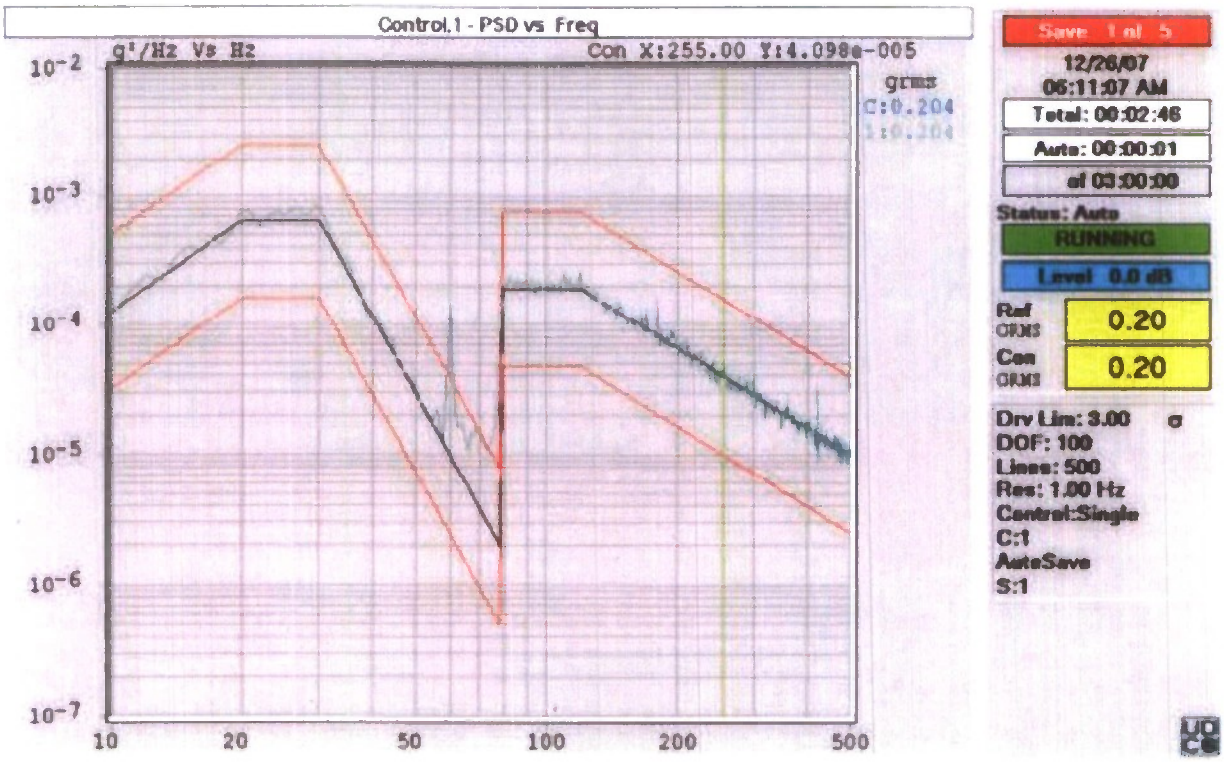


Random Test Report

SETUP NAME: Mil-Std-810F_Trans_CC
SETUP DESC: Mil-Std-810F Common Carrier Transverse Axis
RUN NAME: **GASMET Trans Dec 07**
USER/PROJECT FOLDER: MIL-STD-810F
SAVE NUMBER: 1

STATUS INFORMATION

TEST EVENT TIME: Wednesday, December 26, 2007 at 06:11:07 AM
TEST STATUS: RUNNING
TEST MODE: AUTO
TOTAL TIME ELAPSED (HH:MM:SS): 00:02:46
AUTO TIME ELAPSED (HH:MM:SS): **00:00:01**
TEST LEVEL: 0.0 dB
REFERENCE: 0.20 g rms
CONTROL: 0.20 g rms

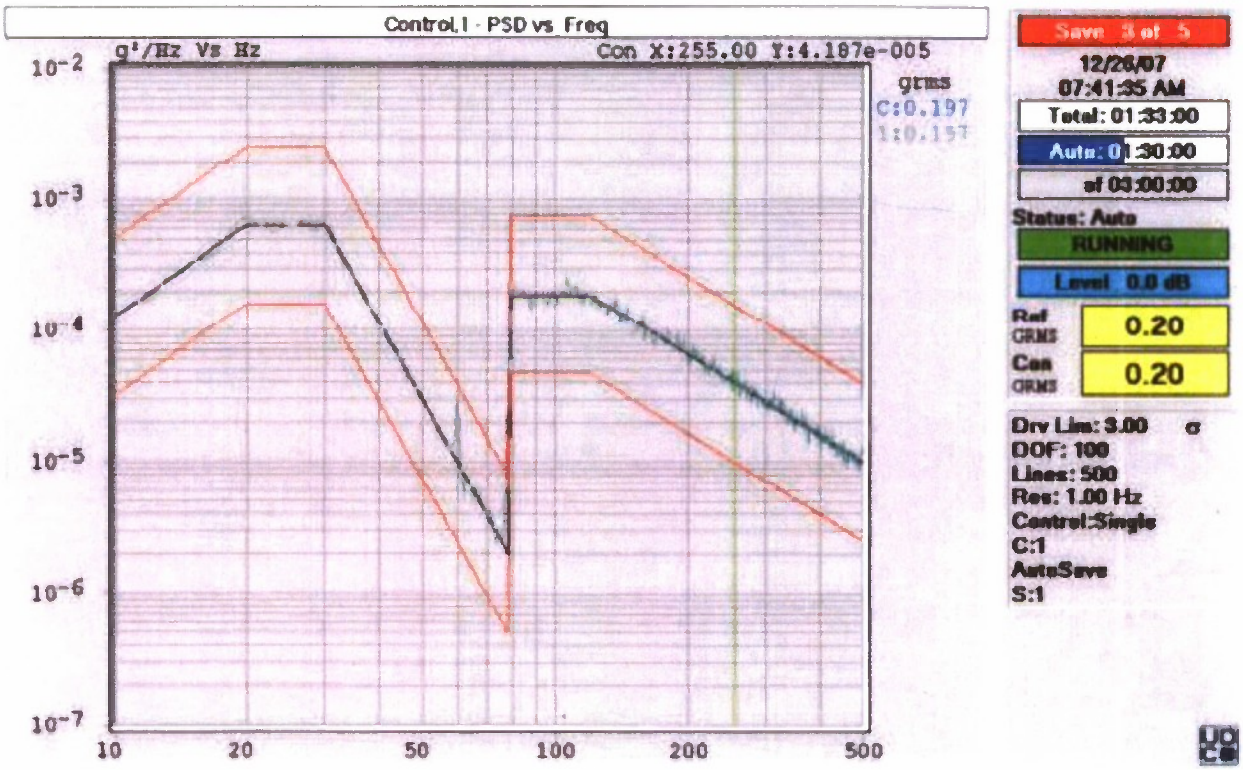


Random Test Report

SETUP NAME: Mil-Std-810F_Trans_CC
SETUP DESC: Mil-Std-810F Common Carrier Transverse Axis
RUN NAME: **GASMET Trans Dec 07**
USER/PROJECT FOLDER: MIL-STD-810F
SAVE NUMBER: 3

STATUS INFORMATION

TEST EVENT TIME: Wednesday, December 26, 2007 at 07:41:35 AM
TEST STATUS: RUNNING
TEST MODE: AUTO
TOTAL TIME ELAPSED (HH:MM:SS): 01:33:00
AUTO TIME ELAPSED (HH:MM:SS): **01:30:00**
TEST LEVEL: 0.0 dB
REFERENCE: 0.20 g rms
CONTROL: 0.20 g rms

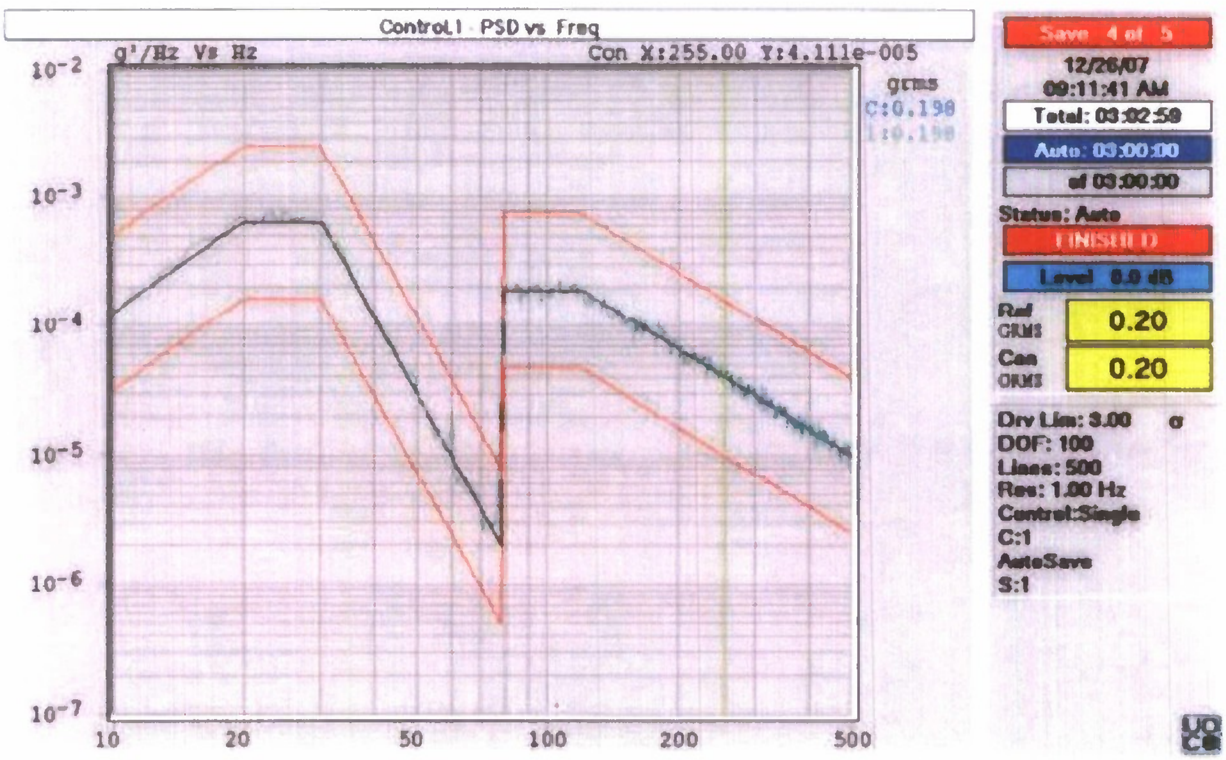


Random Test Report

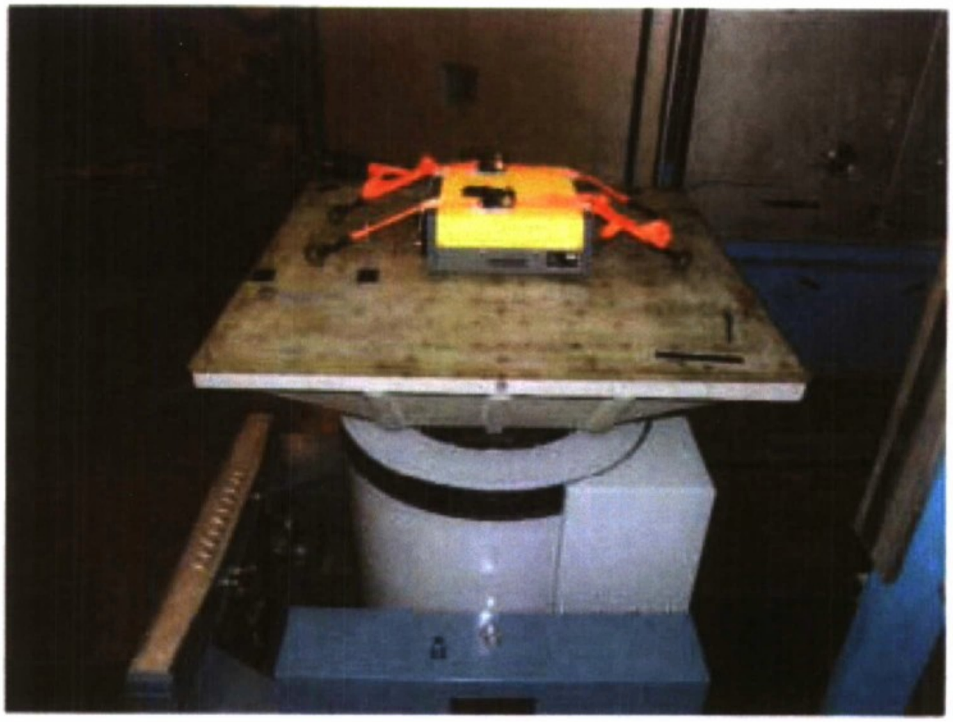
SETUP NAME: Mil-Std-810F_Trans_CC
SETUP DESC: Mil-Std-810F Common Carrier Transverse Axis
RUN NAME: **GASMET Trans Dec 07**
USER/PROJECT FOLDER: MIL-STD-810F
SAVE NUMBER: 4

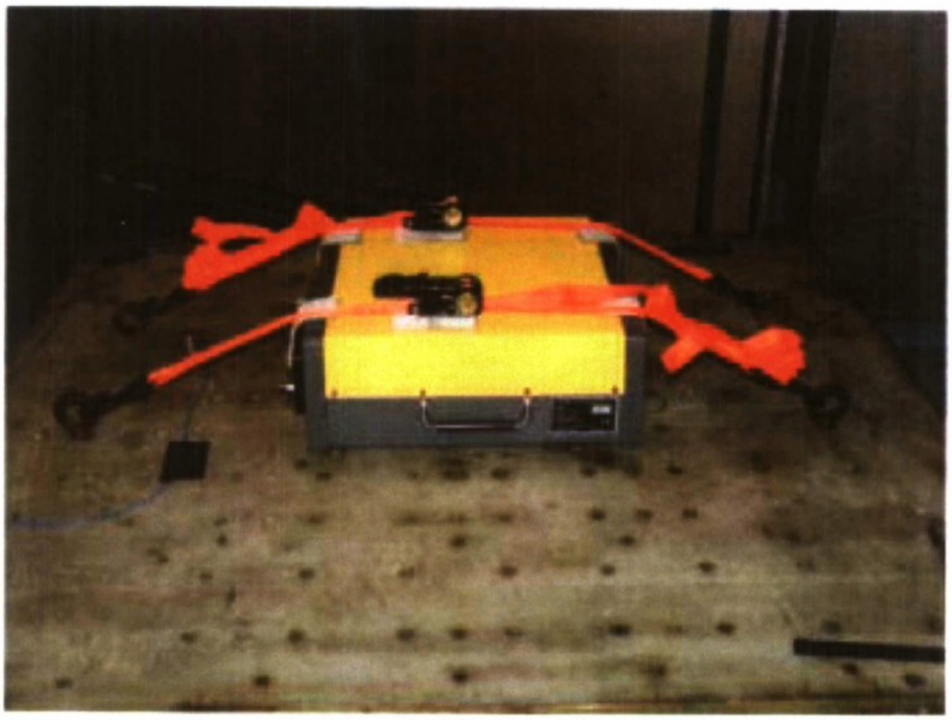
STATUS INFORMATION

TEST EVENT TIME: Wednesday, December 26, 2007 at 09:11:41 AM
TEST STATUS: FINISHED
TEST MODE: AUTO
TOTAL TIME ELAPSED (HH:MM:SS): 03:02:59
AUTO TIME ELAPSED (HH:MM:SS): **03:00:00**
TEST LEVEL: 0.0 dB
REFERENCE: 0.20 g rms
CONTROL: 0.20 g rms



Vertical Axis



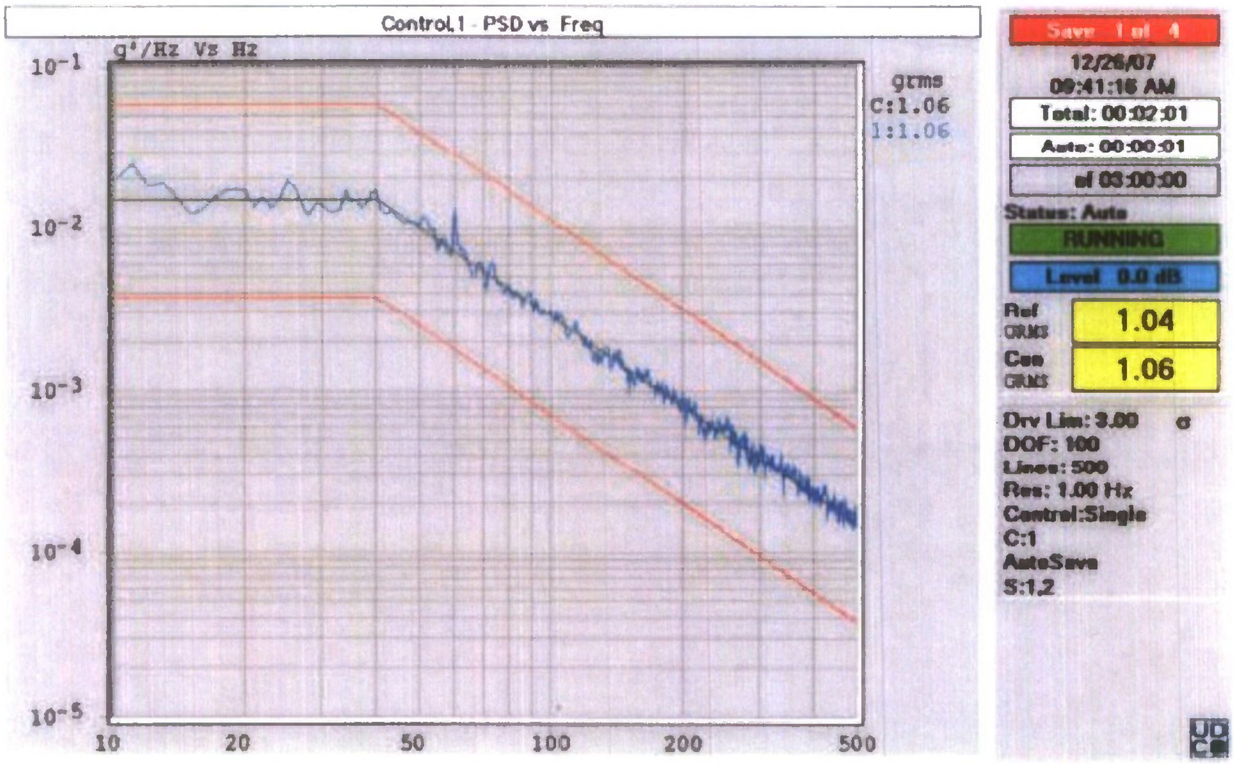


Random Test Report

SETUP NAME: Mil-Std-810F_Vertical_CC
SETUP DESC: Mil-Std-810F Common Carrier Vertical Axis
RUN NAME: **Gasmet Vert Dec 07**
USER/PROJECT FOLDER: MIL-STD-810F
SAVE NUMBER: 1

STATUS INFORMATION

TEST EVENT TIME: Wednesday, December 26, 2007 at 09:41:16 AM
TEST STATUS: RUNNING
TEST MODE: AUTO
TOTAL TIME ELAPSED (HH:MM:SS): 00:02:01
AUTO TIME ELAPSED (HH:MM:SS): **00:00:01**
TEST LEVEL: 0.0 dB
REFERENCE: 1.04 g rms
CONTROL: 1.06 g rms

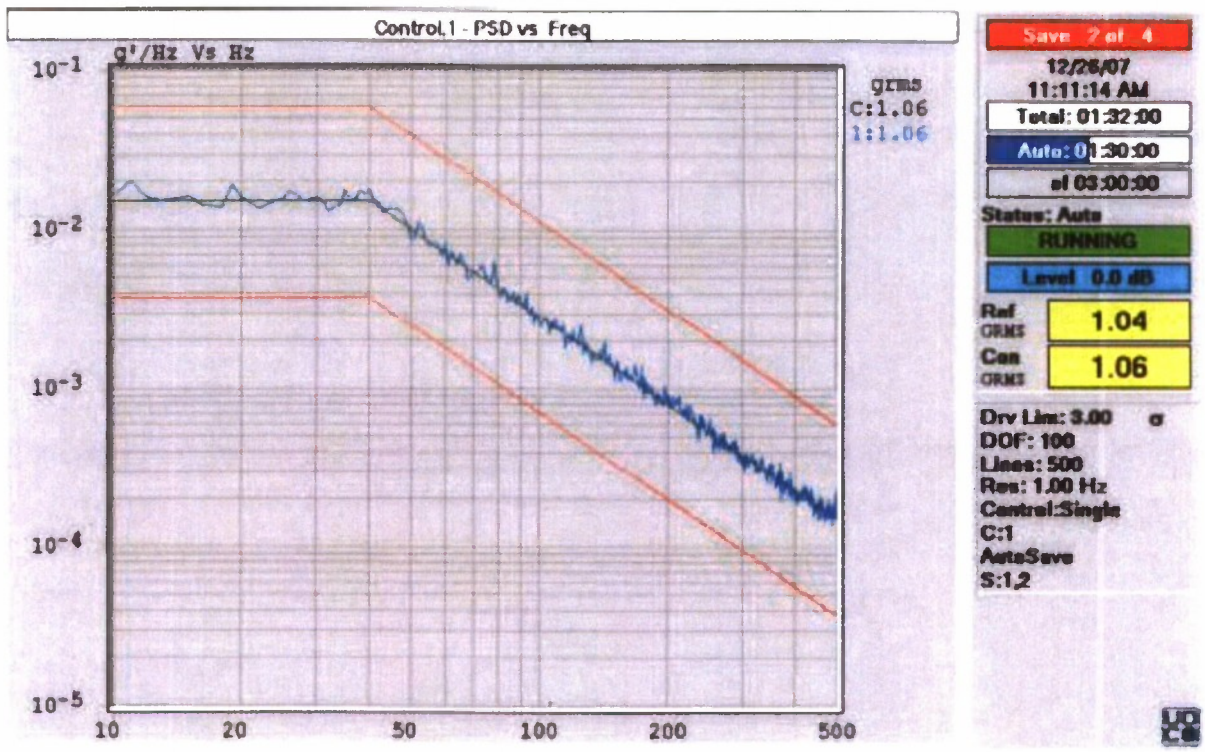


Random Test Report

SETUP NAME: Mil-Std-810F_Vertical_CC
SETUP DESC: Mil-Std-810F Common Carrier Vertical Axis
RUN NAME: **Gasmet Vert Dec 07**
USER/PROJECT FOLDER: MIL-STD-810F
SAVE NUMBER: 2

STATUS INFORMATION

TEST EVENT TIME: Wednesday, December 26, 2007 at 11:11:14 AM
TEST STATUS: RUNNING
TEST MODE: AUTO
TOTAL TIME ELAPSED (HH:MM:SS): 01:32:00
AUTO TIME ELAPSED (HH:MM:SS): **01:30:00**
TEST LEVEL: 0.0 dB
REFERENCE: 1.04 g rms
CONTROL: 1.06 g rms



Random Test Report

SETUP NAME: Mil-Std-810F_Vertical_CC
SETUP DESC: Mil-Std-810F Common Carrier Vertical Axis
RUN NAME: **Gasmet Vert Dec 07**
USER/PROJECT FOLDER: MIL-STD-810F
SAVE NUMBER: 3

STATUS INFORMATION

TEST EVENT TIME: Wednesday, December 26, 2007 at 12:41:20 PM
TEST STATUS: FINISHED
TEST MODE: AUTO
TOTAL TIME ELAPSED (HH:MM:SS): 03:02:05
AUTO TIME ELAPSED (HH:MM:SS): **03:00:00**
TEST LEVEL: 0.0 dB
REFERENCE: 1.04 g rms
CONTROL: 1.07 g rms